Study of Deformation and Erosion Behaviour of Epoxy-Glass Microballoon Based Syntactic Foam

A THESIS SUBMITTED IN PARTIAL FULFILLMENT
OF THE REQUIREMENTS FOR THE DEGREE OF

Master of Technology

In

Ceramic Engineering

By

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Department of Ceramic Engineering
National Institute of Technology
Rourkela-769008, Orissa, India
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CERTIFICATE

This is to certify that the work in the thesis entitled, "**Study of Deformation and Erosion Behaviour of Epoxy-Glass Microballoon Based Syntactic Foam"** submitted by **Mr. Dhirendra Kumar**in partial fulfilment of the requirements for the award of **Master of Technology Degree** in the Department of **Ceramic Engineering**, National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the work reported in this thesis is original and has not been submitted to any other Institution or University for the award of any degree or diploma.

He bears a good moral character to the best of my knowledge and belief.

Arindam Paul

Place: NIT Rourkela
Date: 30.05.15

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For each and every new activity in the world, the human being needs to learn or observe from somewhere else. The capacity of learning is the gift of GOD. To increase the capacity of learning and gaining the knowledge is the gift of GURU or Mentor. That is why we chanted in Sanskrit "*Guru Brahma Guru Vishnu Guru DevoMaheswara, Guru SakshatParam Brahma Tashmey Shree GuruveNamoh*". That means the Guru or Mentor is the path to your destination.

The author first expresses his heartiest gratitude to his guide and supervisor *Prof. Arindam Paul, Assistant* Professor of Ceramic Department for his valuable and enthusiastic guidance, help and encouragement during the course of the present research work. The successful and timely completion of the research work is due to his constant inspiration and extraordinary vision.

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DHIRENDRA KUMAR

ABSTRACT

The present work is focused on the synthesis and study of the deformation behavior of epoxy resin/glass microballoonbased syntactic foams. Different densities syntactic foams with 0 to 40 volume percentages of glass microballoons were preparedby stir-casting method for the present investigation. The high viscosities of the resin-microballoons mixture (putty like consistency) beyond 40 volume percentage prevents processing of higher microballoon content syntactic foam. The effect of glass microballoon content on tensile, compression, flexural and impact properties were studied in detailes. The results show that specific tensile strengths of the foam was increased by about 34% along with the reduction in density (by about 38%), starting from pure resin to foams with 40% glass microballoon. It is also found that compressive strength of the foam decreases from 140 MPa (for pure resin) to 75 MPa (with 40 vol.% reinforcement). During tensile loading, deformation occurs predominantly through shear yielding of the resin matrix followed by debonding at the matrix-microballoon interface. Whereas he crushing of the glass microballoons and subsequent densification of the foams is responsible for large amount of plastic strain during compressive deformation. The erosion behavior of the syntactic foam is also investigated as a function of the different glass microballoons content. Three different erodent velocities (48, 70, 82 m sec^{-1}) and three different angles of impingement (30^o, 60^o and 90^o) are used as experimental parameters in the present investigation. The erosion rate is found to be highest for 40 vol.% reinforcementas compared to pure resin due to presence of larger amount of hollow glass microspheres.

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Chapter -1

Introduction

1.1 Syntactic foam and glass micro balloon

Syntactic foams are closed cell composite material mixed in a resinous matrix which could be thermosetting or thermoplastic. It is a multi functional composite due to its good mechanical property, insulation property, low moisture absorption capacity, low thermal expansion etc. With respect to other process syntactic foam can be easily formed and investment for casting is low. Initially it was developed for sea application due to its light weight, now its application in spacecraft and ship structure. Syntactic foams have advantage over conventional open cell structure, honey comb structures.Syntactic foam are used as core material as a sandwich structure in composite. Conventional core materials are either open cell interconnected porosity or discontinuous in structure. In case of damage to skin in conventional core material water absorption is high then syntactic foam.

To achieve desirable property hollow glass micro balloon will be the good option due to its property like low density, low weight, excellent thermal insulation, low moisture absorption etc. Hollow glass micro balloon is soda lime borosilicate glass having hollow or inert gas inside it.

1.2 Role of matrix in composites

Materials in particulates form or fibrous form shows very good strength but to achieve this strength they should bond with a matrix. The matrix isolates materials to prevent abrasion between them and form a layer act as a new surface or bridge to hold particles. Matrix poses ability to deform easily under applied load, transfer load to reinforcement and distribute stress concentration.

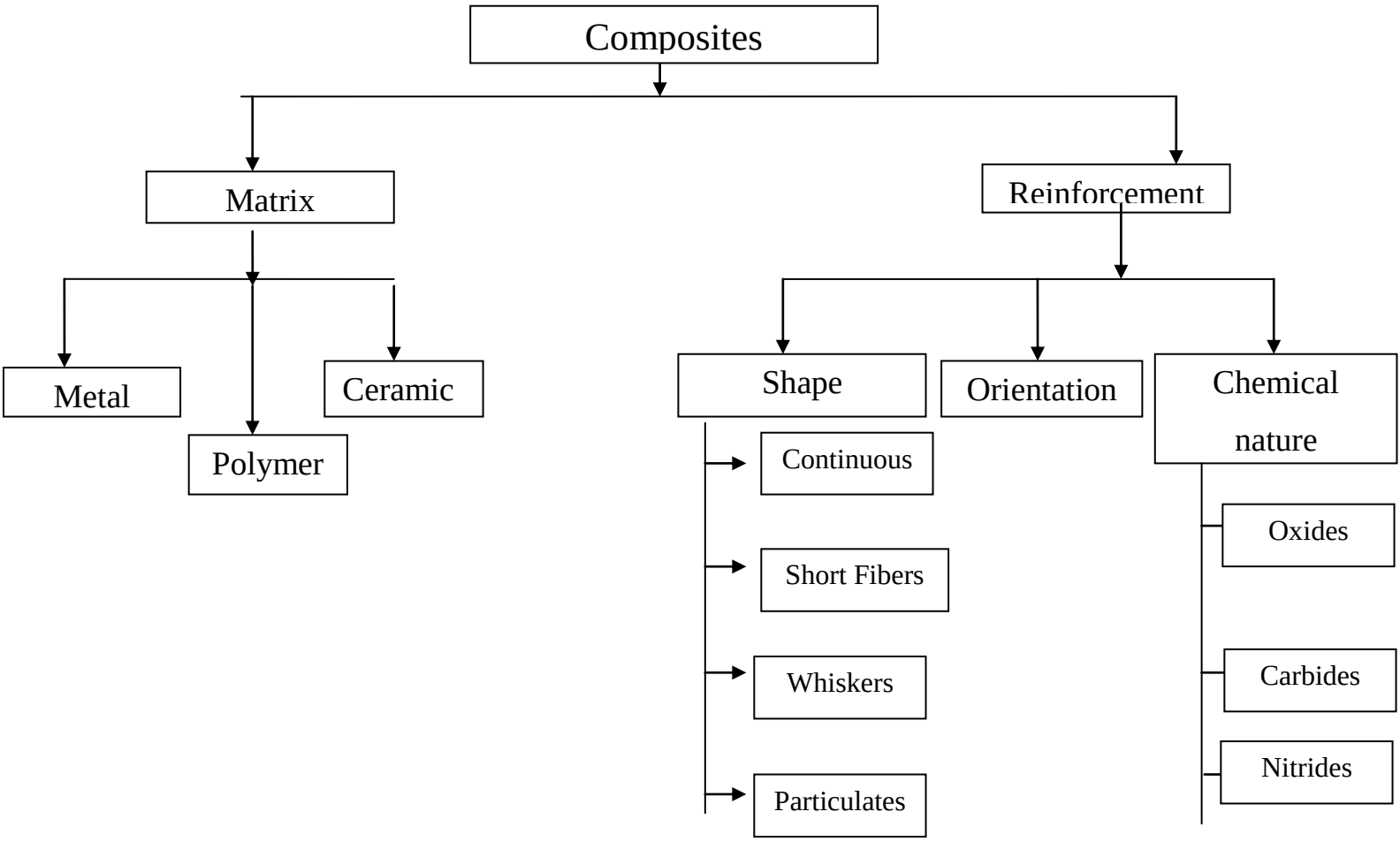
Composites material is composed of at least two materials working together and produces properties different from with their own properties. Practically composites consist of bulk material (matrix) and reinforcement added to increase strength of matrix

1.3 Classification of composites

Composites can be classified on the following basis:

1) On the basis of matrix used
    a) Metal matrix composites
    b) Polymer matrix composites
    c) Ceramic matrix composites

2) On the basis of reinforcement
    a) On the basis of shape
    b) On the basis of orientation
    c) On the basis of chemical nature



1.4 Application of hollow glass microballoon syntactic foam

Marine applications

Top Buoy of Oceanographic mooring

Aerospace applications

Aerospace putty

Fig-1.1 Application of syntactic foam

Chapter 2

Literature Review

2.1 Effect of glass microballoons content on Tensile strength of the syntactic foam

Bibin John *et. al*. [1] worked on the effect of low density reinforcement on mechanical properties of syntactic foam of made of cynate ester.The authors used two types of glass micro balloons having grade of K25 and K37 and performed different testing. From the study the authors concluded that the tensile strength of the syntactic foam were decreased by increasing volume fraction but the syntactic foam with K 37 shows high tensile strength than the K 25 except at higher volume percent. This is the evidence of higher resin to micro balloon bonding in K 37. On increasing volume fraction of the glass microspheres, interfacial bonding between matrix and filler (viz., glass microspheres) decreases and results in decrease in tensile strength.

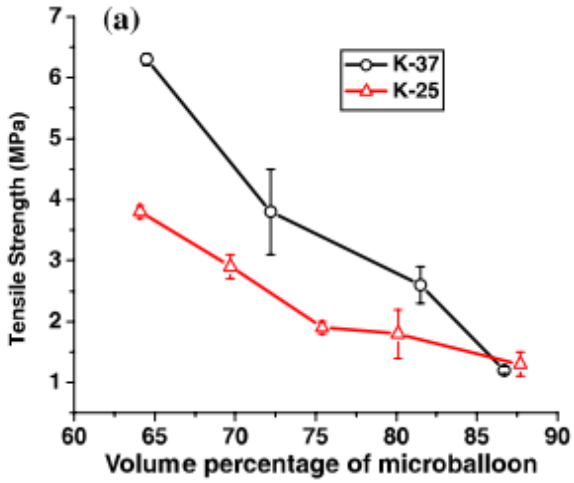


Fig.2.1Variation of tensile strength with different volume content of micro balloon [1]

Erwin M Woutersonet. *al*. [2] studied on specific properties and fracture toughness of syntactic foam: effect of foam microstructure Authors concluded that the increase in glass microsphere and radius to thickness ratio increase specific tensile strength up to 10 volume % and beyond that trend decreases. This is due to the fact that inclusion of hollow microsphere reduces the resin volume fraction and interfaces between glass microsphere and resin decrease, leads to poor bonding between them.

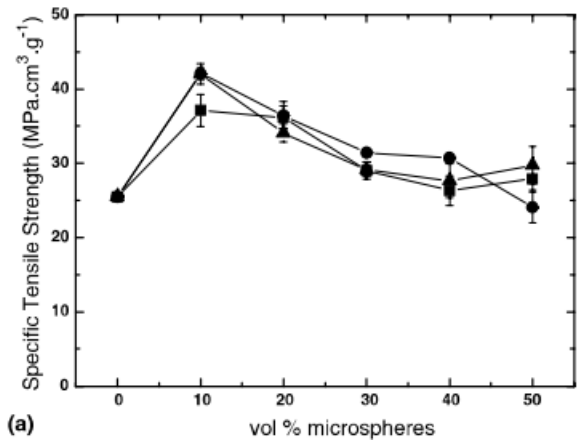


Fig.2.2 Variation of specific tensile strength with volume percentage [2]

Nikhil Gupta*et.al*.[3] studied Comparison of tensile and compressive characteristics of vinyl ester/glass microballoon syntactic foams for different density of glass microsphere 220,320,370,460 kg/m^3 Authors observed that the Young's modulus of composition was greater than vinyl ester resin and specific modulus of all volume % is greater than neat epoxy. This is due to the porosity present in glass microsphere and it was high in higher volume fraction.

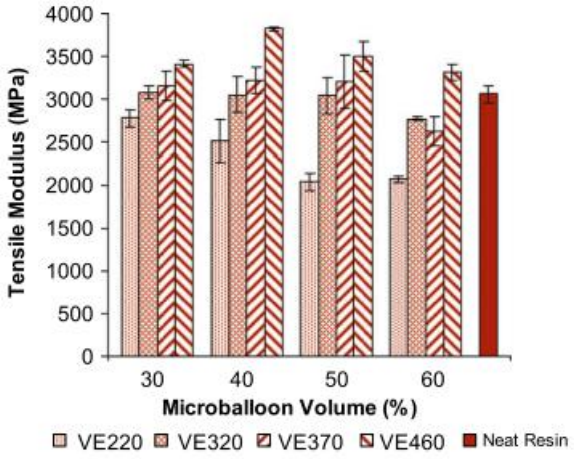


Fig. 2.3 Variation of tensile modulus with volume % of glass micro balloon [3]

2.2 Effect of glass micro spheres content on compressive behaviour of the syntactic foam

Bibin John *et. al*. [1] worked on the effect of low density filler on mechanical properties of syntactic foam of cynate ester Authors conclude that the compressive strength and specific compressive strength shows a slow decrease with increase in volume fraction of glass micro

balloon. Compressive strength depends on void content, volume fraction, and wall thickness of glass micro balloon. With increase in volume fraction of glass micro balloon, the micro balloon takes up more loading under compression and load bearing capacity of glass balloon is not well. With increase in volume percentage the layer between matrix and glass balloon decrease and fracture easily under stress.

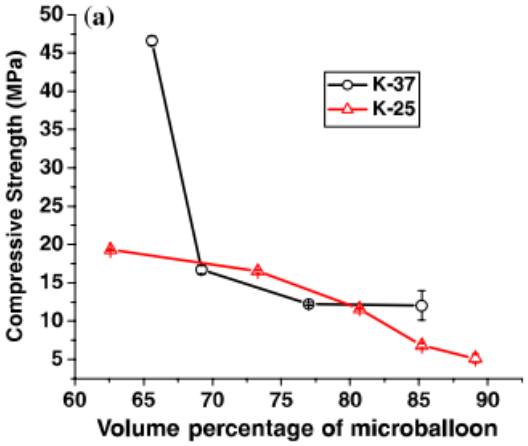


Fig.2.4 Compressive strength and volume percantage relation[1]

C. Sweta and R Kumar. [4] worked on the quasi static uniaxial behavior of hollow glass microsphere/epoxy based syntactic foam. Authors used different densities HGMs and were prepared by stir casting method in order to find mechanical property. They also investigated effect of volume percentages of hollow glass sphere and wall thickness on the mechanical behavior.

From their experiment they made following conclusion:

1) Compressive strength and modulus decrease with increase in wall thickness as well as increase in volume fraction of micro balloon.

2) Nature of stress –strain curve is stable or unstable is depend on the wall thickness of sphere.

3) With increase content of hollow glass microsphere energy absorption capacity of foam is increase and strength decrease until critical volume fraction.

4) Failure occurs by shear as well as axial splitting and deformation behavior is similar to cellular solids.

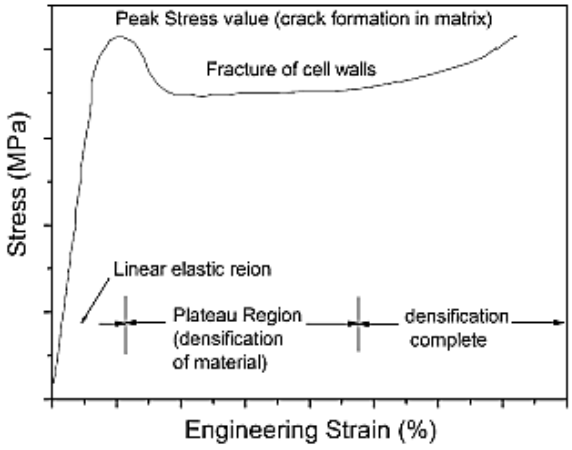


Fig.2.5 Different region involved during compression test [5]

Xie Wenfeg*et.al* [6] studied the Compressive and Fracture Properties of Syntactic Foam filled with Hollow Plastic Bead(HPC). Authors observe that the compressive strength of syntactic foam decreases with increase in plastic beads content this is because with increase in plastic beads the interfaces between matrix and beads increases which decreases the compressive strength of composite. The other reason may be air bubbles introduce in composite which reduce the properties of the syntactic foam.

Ph Viot *et. al.* [7] studied the effect of strain rate and density on dynamic behavior of syntactic foam. Authors observed that the volume fraction of glass sphere plays main role in compressive characteristics like compressive modulus and compressive strength. An increase in strain rate was increasing compressive modulus and compressive strength.

Nikhil Gupta *et. al*. [8] studied the enhancement of energy absorption in syntactic by nanoclay incorporation for sandwich core application. Authors observed that the compressive strength was decreased by incorporation of 2% nanoclay by volume, and 5 % nanoclay give same level strength as compared to 2%.

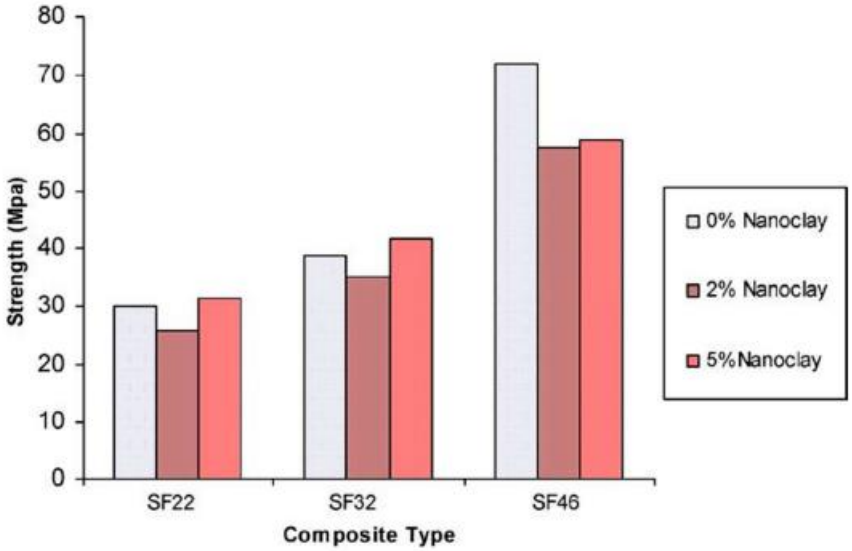


Fig.2.6 Comparison of compressive strength with different volume percent of nanoclay[8]

2.3 Effect of glass microballoons content on flexural strength of the syntactic foam

Bibin John *et. al.*[1] studied on the effect of low density filler on mechanical properties of syntactic foam of cynate ester Authors concluded that the flexural strength and specific flexural strength decrease with increasing volume fraction. This is due to the fact that with increase in volume fraction the layer between matrix and surrounding glass microballoon becomes thin. Therefore the flexural strength decreases due to easy breaking of thin film between matrix and glass micro balloon.

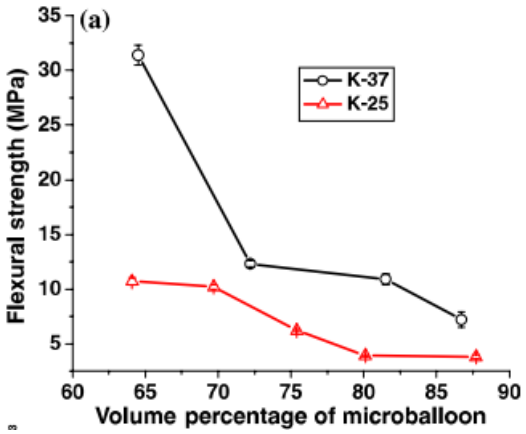


Fig.2.7 Flexural strength vs volume percent relation[1]

Erwin M Wouterson *et. al*. [2] studied on specific properties and fracture toughness of syntactic foam: effect of foam microstructure authors observe that the all type of microsphere shows decrease in specific flexural with increase in volume fraction this is due to debonding of microsphere at higher volume percentage. Debonding shows poor interface between matrix and fillers.

UlkuYilmazer *et.al*. [9] studied on Tensile, flexuraland impact properties of a thermoplastic matrix reinforced by glass fiber and glass bead hybrids authors observed that flexural strength decreases with increase in filler content this is due to *vacuole growth* . Vacuole in the flexural test was developed in the tension side of the specimen.

2.4 Effect of glass micro spheres content on Impact behaviour of the syntactic foam

Ho Sung Kim*et. al*.[10] work on fracture and impact behavior of hollow microsphere/epoxy resin composite authors observed that the impact strength of composite decrease with increase in volume percentage of glass micro balloon this was due to the breakage of specimen at high volume fraction and strength of glass balloon was less.

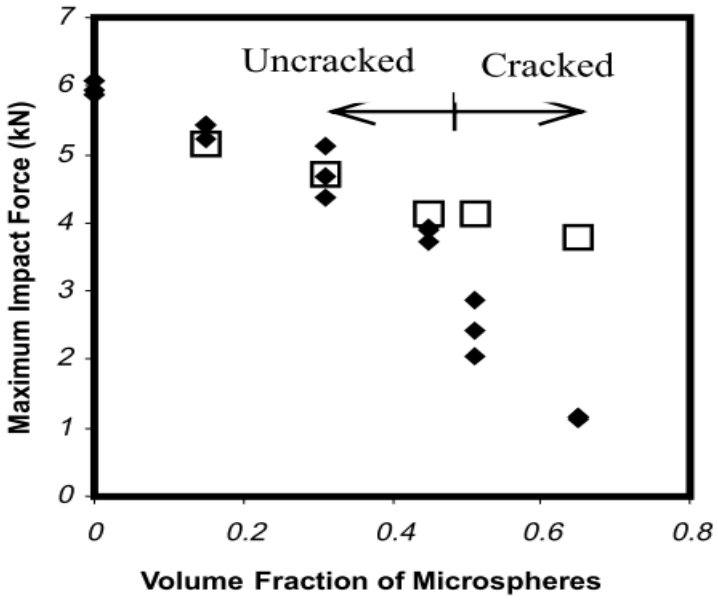


Fig. 2.8 Variation of impact force with volume fraction of microsphere[10]

Witold Brostow *et.al*.[11] studied Brittleness of materials: implications for composites and a relation to impact strength authors observe that adding of ceramic particle may change polymer composite brittleness. It may increases or decreases depend on the particle dispersion filler matrix adhesion and the amount of filler added.

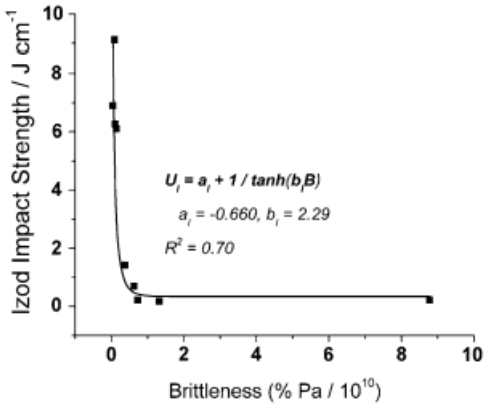


Fig.2.9 Variation of impact strength with percentage brittleness [11]

Ph Viot*et.al*.[7] studied on the effect of strain rate and density on dynamic behavior of syntactic foam authors observed that energy absorbed for syntactic foam for impact test was depend on two factors first was height of the fall and mass of the projectile second was volume fraction of glass microsphere. Mass of the sample had not put any effect on impact energy. Glass microsphere was significant for higher volume fraction of microsphere and epoxy resin significant for lower volume fraction of microsphere.

UlkuYilmazer *et.al.* [9] studied on tensile, flexural and impact properties of a thermoplastic matrix reinforced by glass fiber and glass bead hybrids on notched and unnotched sample Authors observed that impact energy decrease with increase in volume fraction of glass beads because vacuole growth in impact test is absent and at high speed extensibility of matrix was reduced.

2.5 Effect of glass micro spheres content on Hardness behaviour of the syntactic foam

Xiao-Feng Li*et. al*.[12] studied Mechanical properties of epoxy-based composites using coiled carbon nanotubes authors observed that the hardness of the coil carbon nanotubes increase with increase in weight percent of the coil carbon nano tubes this was due to the fact that coil carbon nano tubes dispersed and interlock well in the epoxy.

Chun-Ki Lam *et. al*.[13] studied Cluster size effect in hardness of nanoclay/epoxy composites authors observed that hardness of composites increase with increase in nanoclay up to a optimum level and then decreasing, this was due to the cluster size of nano clays reaches a crucial limit and the reinforcing function of clay decrease with increase in nano clay in composite.

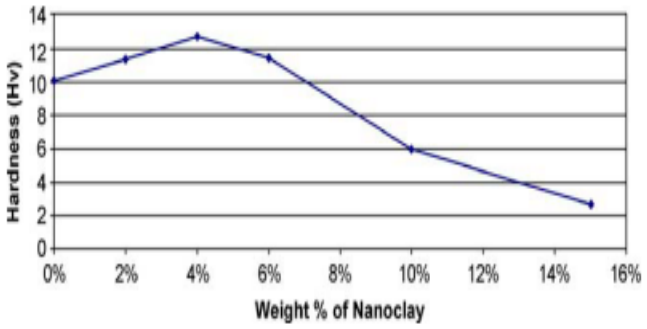


Fig.2.10 Variation of hardness with weight % of nano clays[13]

Qi Tang *et.al*.[14] studied the Effect of porosity on the microhardness testing of brittle ceramics: A case study on the system of NiO – ZrO_2 authors observed that for different porosity 2% to 18 % for every sample the hardness value decrease with increase in porosity this was due to the fact that the with increase in porosity decrease in densification occur leads to the decrease in resistant to indentation .

2.6 Effect of content of glass micro spheres content on Erosion behaviour of the syntactic foam

G Raghvendra*et. al.* [15] studied A Comparative Analysis of Woven Jute/Glass Hybrid Polymer Composite With and Without Reinforcing of Fly Ash Particles authors observed that glass fiber had less bonding nature than jute fiber jute outer layer behaves as brittle because it

shows maximum erosion at 90^0. After addition of fly ash composite behavior was same and it shows maximum at 60^0 which indicate semi brittle behavior of composite.

G Raghvendra*et. al.* [16] studied the Studying the Parameters of the Solid Particle Erosion and Test Procedure.The authors found out pressure and velocity relation for erosion testing machine by rotating disc method the relation was as follows:

Table.2.1.Relationship between pressure and average impact velocity in air jet erosion[16]

Pressure (bar)	Speed of rotating disc(rpm)	Angle θ (°)	Velocity(m/s)	Avg. impact velocity(m/s)
1 bar	2000	7.0	42.85	47.25
		6.5	46.15	
		6.0	50.00	
		6.0	50.00	
		4.0	75.00	
2 bar	2000	4.5	66.67	69.16
		4.0	75.00	
		5.0	60.00	
		4.5	66.67	
		4.0	75.00	
3 bar	2000	3.5	85.71	81.845
		3.0	100.00	

ShakuntalaOjha*et.al*. [17] Studied the Characterization and Wear Behavior of Carbon Black Filled Polymer Composites. Authors observed that for 48 m/s impact velocity maximum wear for neat epoxy at 90^0 impingent angle means neat epoxy shows brittle behavior. When adding raw wood shell then maximum wear occurs at 30^0 to 45^0 for all composites samples means nature of sample brittle to semi brittle shows increase in erosion rate.

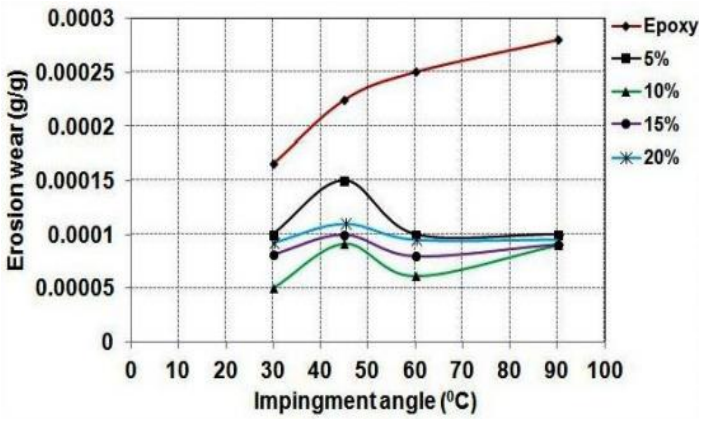


Fig.2.11 Variation of erosion wear with impingement angle [17]

Objectives of the Present work

As discussed in the earlier section, thermoset/thermoplastic polymer-glass microballoon based syntactic foamsare having various attractive mechanical properties and having the potential to cater as modern high performance light weight composite materials. In the present work, study of the different mechanical and erosion properties of epoxy resin-glass microballoon based syntactic foam for structural applications is proposed.

Justification of the work:

- Epoxy resin was chosen as matrix material due to its high strength, low shrinkage, excellent adhesion with fillers, low cost and toxicity.
- Glass microballoonwas selected as filler due to its high compressive strength and stiffness.
- Mechanical and erosion properties of composite foam need to be optimized for structural application.

Aim of the present work:

- To develop light weight, higher strength epoxy-glass microballoonsyntactic foam
- To study deformation behaviour of syntactic foam
- To study erosion behaviour of syntactic foam

Chapter 3

Experimental Procedures

3 Experimental procedures

The aim of the present work is to develop glass microballoon–epoxy based syntactic composite foam and study of their deformation behavior. In this chapter, processes used in order to develop this type of syntactic foam is described first, followed by details description of various characterization techniques.

3.1 Constituents materials

3.1.1 Matrix materials

Epoxy resin (Araldite LY556) and hardener (HY951) manufacturedby Huntsman Advanced Materials India was used as a matrix material and curing agent respectively. Araldite (LY556) was highly viscous; having density and viscosity of 1.15g/cc and 10000-12000 mPa-s respectively. Hardener (HY951) was low viscous having density 0.98g/cc was used for room temperature curing. The resin to hardener ratio used was 10:1.

3.1.2 Reinforcement

Glass micro balloons of grade K15 was supplied by 3M India limited was used as reinforcement for developing syntactic foam. The glass microsphere was chemically stable having composition of soda lime borosilicate glass having thermal stability of 600^0C.

Calculation for wall thickness and radius ratio

Wall thickness and radius ratio can be calculated from the following formula:

Wall thickness of the glass microballoons, $\mathbf{e/r = [1-(1-\rho_{true}/\rho_{glass})^{1/3}]}$**.....**(3.1)

Radius ratio (inner radius to outer radius), $\mathbf{\eta=(r-e)/r}$**.....** (3.2)

e = Wall thickness of glass micro balloon, r = average radius of glass micro balloon (25µm), ρ_{true}= true density of glass micro balloon (0.15g/cc), ρ_{glass}= density of glass (2.54g/cc)

By putting the above value in equation (3.1) and (3.2) we get values of wall thickness and radius ratio as shown in below table.

Table 3.1 Properties of hollow glass microballoon

Property	Reinforcement (K15)
Effective size (μm)	50
Density (g/cc)	0.15
Average wall thickness (μm)	0.50
Radius ratio-η	0.97
Isostatic crush strength (MPa)	2

3.2 Specimen processing

Stir casting method was used, in the present study, to process syntactic foams with varying volume percentage (0-40 volume %) of glass microballoons. Required amount of resin, hardener, and glass microballoons, depending on the volume percentage of the resultant foam were calculated, a priori (as shown below).

3.2.1 Calculation for required amount of constituent materials for different volume percentage of syntactic foam

In this subsection, calculation of required amount constituent materials (e.g. epoxy resin, hardener, and glass microballoon) are described for x volume% of epoxy and y volume% of filler. The required amount of the constituent for other volume percentage is calculated in a similar manner.

Calculation for 5 volume percent

The calculation for weight estimation for matrix and filler are as follow:

Calculation for matrix

Density of epoxy =1.15g/cc

Density of hardener =0.98g/cc

Density of glass micro balloon=0.15 g/cc

Here epoxy to hardener ratio used was 10:1

Percentage of epoxy in matrix= (10/11) ×100=90.9%

Percentage of hardener in matrix= (1/11)×100=9.09%

By applying rule of mixture for obtaining density of matrix

Density of matrix= density of epoxy × volume fraction of epoxy+ density of hardener × volume fraction of hardener

Density of matrix= (90.9/100)×1.15+ (9.09/100)× 0.98

Density of matrix=1.12g/cc

Volume of mould =150×65×5(mm^3) = 48750 mm^3

Let volume percentage of epoxy and glass micro balloon taken was x and y respectively

Density= mass/volume

1.12×10^{-3}= mass/(x×48750)

Mass of matrix= 54.6x g

Mass of epoxy= (90.9×54.6x)/100

Mass of epoxy= 49.63x g

Mass of hardener = (9.09×54.6)/100

Mass of hardener = 4.9x g

Mass of filler

Density of filler= mass/volume

0.15×10^{-3}= mass/(y×48750)

Mass of filler= 7.312y g

Similar manner it was calculated in respective places for different tests

3.2.2 Preparation of Mold

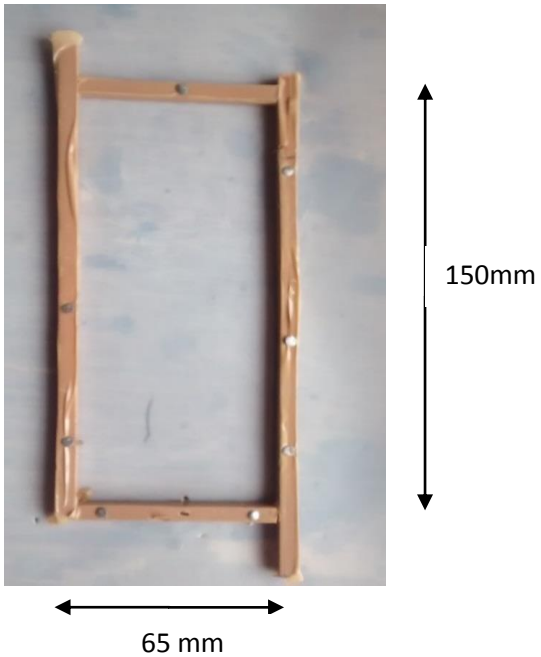


Fig. 3.1 Prepared mould made from wooden bit

In the present study the dimension of the mould used to prepare specimen for tensile and flexural testing is 150mm×65mm×7mm. At first wooden bits (of thickness 7.0mm) were cut according to the dimension of 150mm and 65mm respectively. Then these wooden bits were wrapped with plastic tape.

A flat wooden board fixed with a transparent plastic sheet was used to form the base of the mold. The wrapped wooden bits were fixed firmly (with the help of nails) in rectangular shape as shown in figure 3.1. The purpose of the transparent plastic sheet is to prevent sticking of the mixture and ease removal of the specimen after room temperature curing.

3.2.3 Preparation of syntactic foam

The flow sheet for preparation of the syntactic foam is shown in figure 3.2.

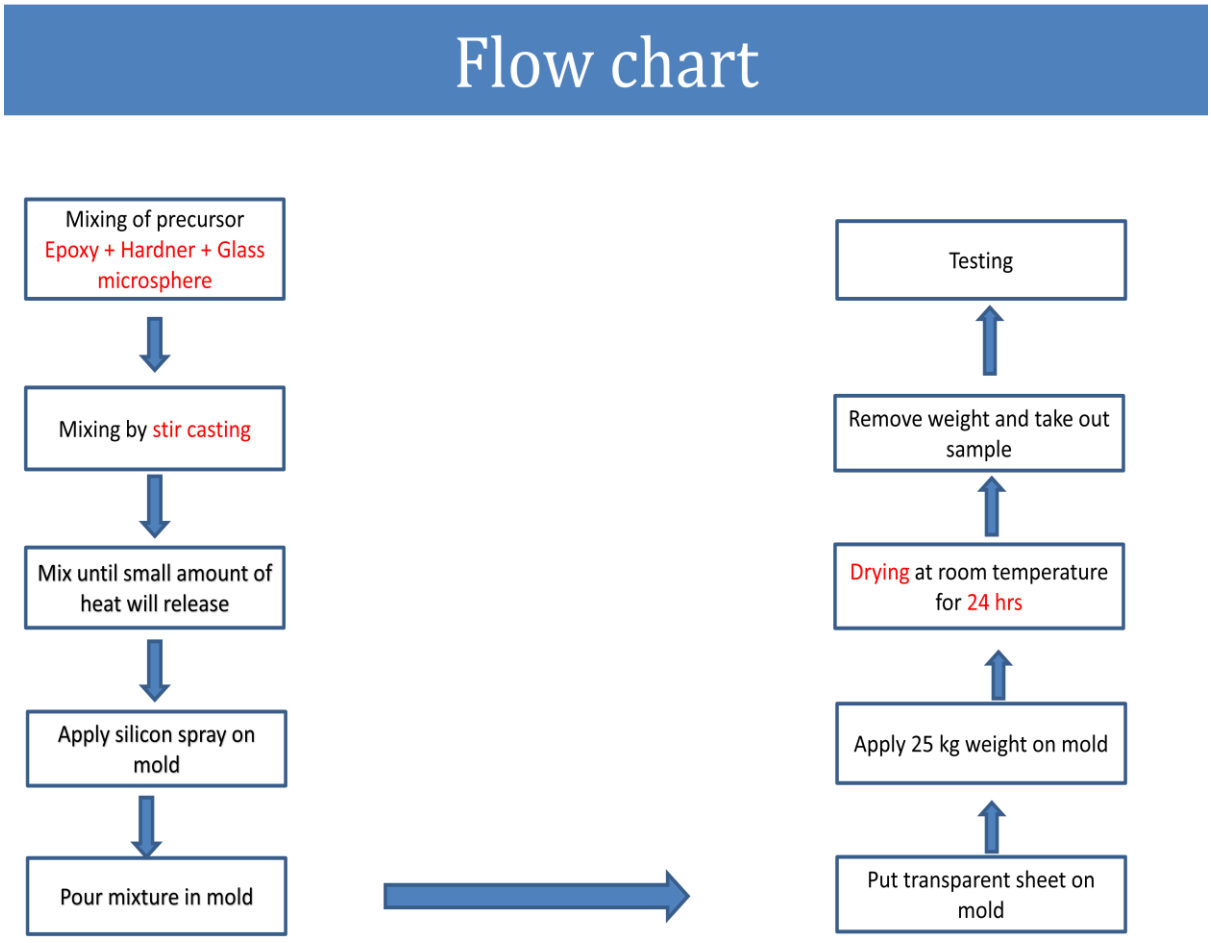


Fig. 3.2 Flow chart of the syntactic foam processing

Calculated amount of epoxy, hardner were weighed first and was taken in a plastic beaker. The solution was then mixed by manual stirring using a glass rod till small amount of heat was released; this release of heat is an indication of exothermic reaction between hardner (i.e. catalyst) and epoxy that leads to cross-linkning of polymers. After that required amount of glass micro balloon was added gently to the resin- hardener mixture. Enough care was taken during the slow manual stirring, which was preferred in order to avoid breakage of glass microballoons. Since glass microballoons are less dense compared to the resin mixture, they may tend to segregate during the mixing process and float on the top. Gentle mechanical stirring was continued for 12-15 min to ensure uniform distribution of microballoon in the resin matrix.

With the increase in microballoon content, the viscosity also increases and the resin-microballoon mixture has putty like consistency. Beyond 40 volume % of glass microballoon (maximum volume percentage used in the present study), the viscosity of the mixture became so high, it was difficult to process it further.

The mixture was then transferred to the rectangular wooden mould (shown in figure 3.1), which was smeared with silicone gel (mould releasing agent). Then a transparent plastic sheet was placed on top of the mixture. The sample along with the mould was cured at room temperature for 24 hours under approximately 25 kg load. The different steps of the syntactic foam processing is shown in fig. 3.3.

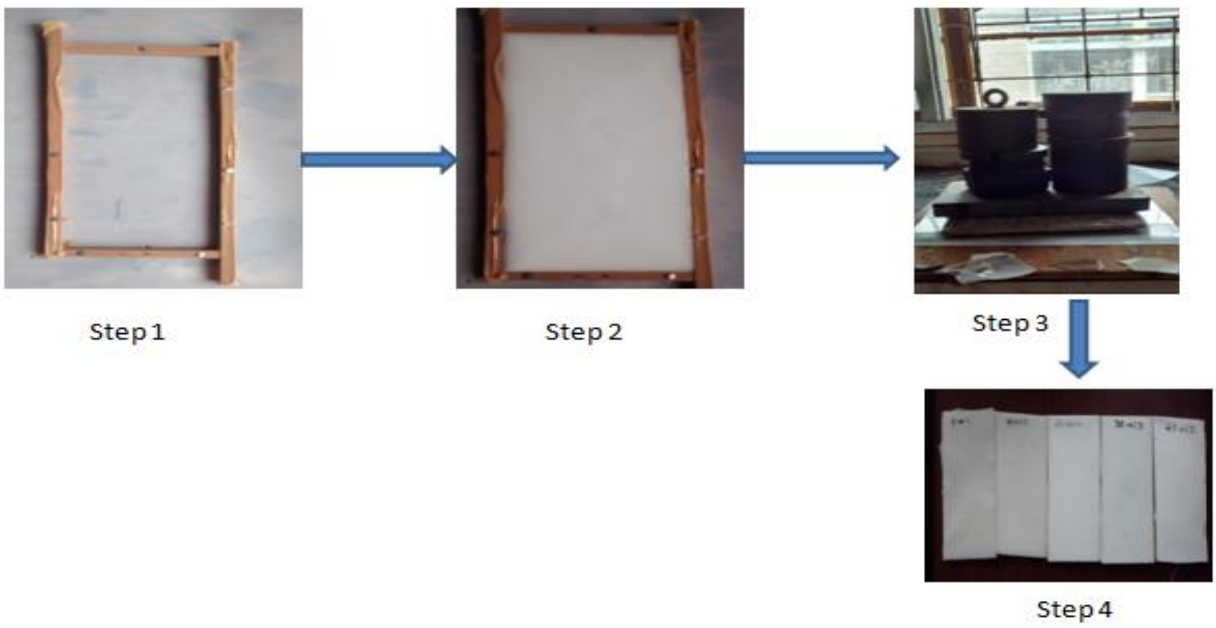


Fig 3.3 Steps involves for processing of syntactic foam

3.3 Physical characterization

3.3.1 Density measurement

The density of syntactic foam was calculated by the formula (rule of mixture) as given in equation (3.3) and density of the foam specimen was measured by Archimedes density measurement kit as given in equation (3.4).

$\rho_c = p_{m\times}v_m + \rho_{f\times}v_f$.....(3.3)

$\rho_c = [w_a(\rho_l-\rho_a)/0.999(w_a - w_l)]+\rho_a$.....(3.4)

Where

ρ_c= density of composite (g/cc), ρ_f, ρ_m-density of glass micro balloon and matrix (g/cc).v_f,v_m-volume fractions of microballoon and matrix.

ρ_a-density of air(0.0012g/cc),w_a, w_l, weight of sample in air and liquid respectively.

3.3.2 X- ray diffraction studies (XRD)

Rigaku model ultima-IV was used for x-rays analysis of microballoon. The wavelength used for scanning was Cu Kα. The scan rate, step size and scan range used for XRD were 2^0/min, step size 0.005 and scanning range 10^0 to 80^0respectively.

3.3.3 Particle size analyser

Malvern particle size analyzer was used to find out particles size of glass microballoon. The glass micro balloon was ultrasonicated with water for 10 minute before putting inside the machine to remove soft agglomerate.

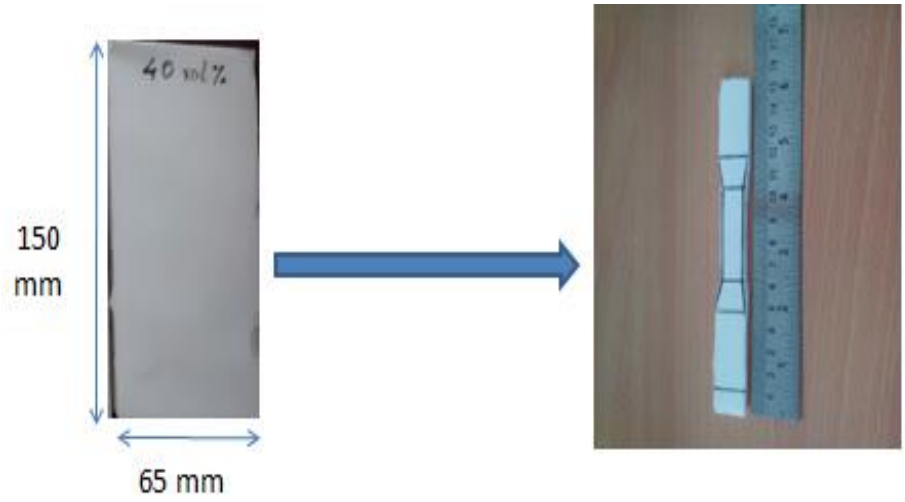
3.3.4 Microstuctural analysis

The fracture surfaces were analyzed by using FEG-SEM (NOVA NANO SEM, FEI). To avoid electron charge build up, gold coating was sputtered on the fracture surface in priori.

3.4 Mechanical Characterization

3.4.1 Tensile testing

The test was performed on universal testing machine INSTRON H10KS. Tensile test specimens of dog bone shape as shown in figure were prepared according to standard **ASTM E-8/8M.** Thecross head speed was fixed at 1mm/min. Tests were carried out on four samples at each set.



(a) (b)

Fig 3.4 (a) syntactic foam slab, as casted, (b) dog bone shape prepared from as casted slab.

Calculation for tensile specimen

Sample calculation for 0 volume %

The calculation for weight estimation for matrix and filler are as follow:

Calculation for matrix

Density of epoxy =1.15g/cc

Density of hardener =0.98g/cc

Density of glass micro balloon=0.15g/cc

Here epoxy to hardener ratio used was 10:1

Percentage of epoxy in matrix= (10/11) ×100=90.9%

Percentage of hardener in matrix= (1/11)×100=9.09%

By applying rule of mixture for obtaining density of matrix

Density of matrix= density of epoxy × volume fraction of epoxy + density of hardener × volume fraction of hardener

Density of matrix= (90.9/100)×1.15+ (9.09/100)× 0.98

Density of matrix=1.12g/cc

Volume of mould =150×65×7(mm^3) = 68250mm^3

Density= mass/ volume

1.12×10^{-3}= mass/(1×68250)

Mass of matrix= 76.440g

Mass of epoxy= (90.9×76.440)/100

Mass of epoxy= 69.48g

Mass of hardener = (9.09×76.440)/100

Mass of hardener = 6.94g

Sample calculation for 40 volume %

The calculation for weight estimation for matrix and filler are as follow:

Calculation for matrix

Density of epoxy =1.15g/cc

Density of hardener =0.98g/cc

Density of glass micro balloon=0.15g/cc

Here epoxy to hardener ratio used was 10:1

Percentage of epoxy in matrix= (10/11) ×100=90.9%

Percentage of hardener in matrix= (1/11)×100=9.09%

By applying rule of mixture for obtaining density of matrix

Density of matrix= density of epoxy × volume fraction of epoxy+ density of hardener × volume fraction of hardener

Density of matrix= (90.9/100)×1.15+ (9.09/100)× 0.98

Density of matrix=1.12g/cc

Volume of mould =150×65×7(mm^3) = 68250mm^3

Density= mass/ volume

1.12×10^{-3}= mass/(0.6×68250)

Mass of matrix= 45.864g

Mass of epoxy= (90.9×45.864)/100

Mass of epoxy= 41.69 g

Mass of hardener = (9.09×45.864)/100

Mass of hardener = 4.16 g

Mass of filler

Density of filler= mass/ volume

0.15×10^{-3}= mass/(0.40×68250)

Mass of filler= 4.095 g

In the similar manner calculation for all volume % of glass micro balloon was done and given in table:

Table 3.1 Mass of epoxy, hardener, and glass micro balloon

Volume %	Mass of epoxy (gm)	Massof hardener (gm)	Mass of glass micro balloon (gm)
0	69.48	6.94	0
5	66.01	6.60	0.51
10	62.53	6.253	1.023
20	55.58	5.58	2.047
30	48.638	4.863	3.07125
40	41.69	4.169	4.095

3.4.2 Compression test

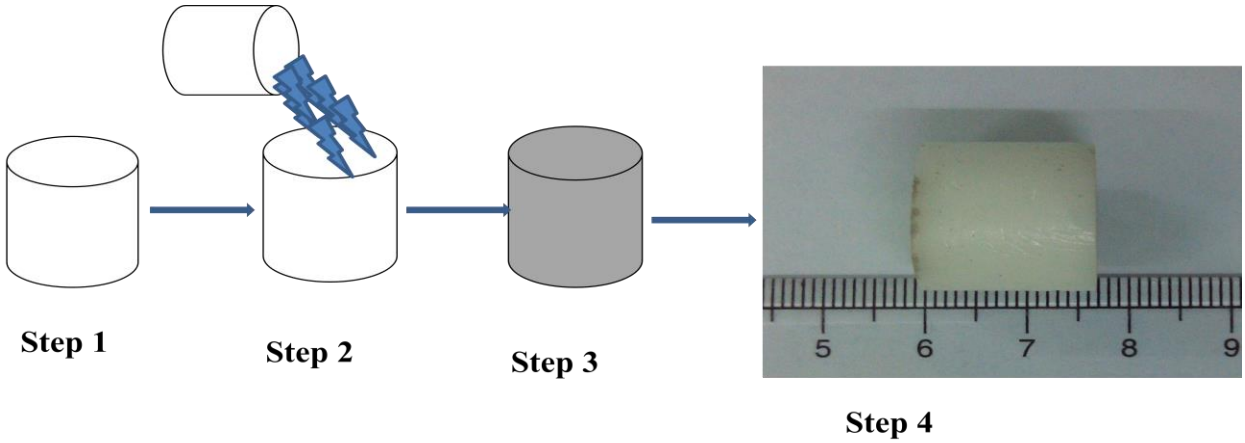


Fig 3.5 Preparation of compression specimen

Compression test specimen of cylindrical in shape having L=17mm and D=17mm approximately were prepared by as per **ASTM standard C365-03**and test was perform by using universal testing machine at a strain rate0.006s^{-1}. Test was performed on UTM Instron model no. BSUT-60-JD supplied BLUE STAR LIMITED - INDIA 2.

Calculation for compression test

Sample calculation for 0 volume %

The calculation for weight estimation for matrix and filler are as follow:

Calculation for matrix

Density of epoxy =1.15g/cc

Density of hardener =0.98g/cc

Density of glass micro balloon=0.15g/cc

Here epoxy to hardener ratio used was 10:1

Percentage of epoxy in matrix= (10/11) ×100=90.9%

Percentage of hardener in matrix= (1/11)×100=9.09%

By applying rule of mixture for obtaining density of matrix

Density of matrix= density of epoxy × volume fraction of epoxy+ density of hardener × volume fraction of hardener

Density of matrix= (90.9/100)×1.15+ (9.09/100)× 0.98

Density of matrix=1.12g/cc

Inner diameter of pipe = 17mm

Height of pipe = 17mm

Volume of cylindrical mould = $(\pi \times 17^2 \times 17)/4(mm^3) = 3856.705mm^3$

Density=mass/ volume

1.12×10^{-3}=mass/(1×3856.705)

Mass of matrix= 4.319 g

Mass of epoxy= (90.9×4.319)/100

Mass of epoxy= 3.926 g

Mass of hardener = (9.09×4.319/100

Mass of hardener = 0.392 g

Sample calculation for 40 volume %

The calculation for weight estimation for matrix and filler are as follow:

Calculation for matrix

Density of epoxy =1.15g/cc

Density of hardener =0.98g/cc

Density of glass micro balloon=0.15g/cc

Here epoxy to hardener ratio used was 10:1

Percentage of epoxy in matrix= (10/11) ×100=90.9%

Percentage of hardener in matrix= (1/11)×100=9.09%

By applying rule of mixture for obtaining density of matrix

Density of matrix= density of epoxy × volume fraction of epoxy+ density of hardener × volume fraction of hardener

Density of matrix= (90.9/100)×1.15+ (9.09/100)× 0.98

Density of matrix=1.12g/cc

Diameter of pipe = 17mm

Height of pipe = 17mm

Volume of cylindrical mould = $(\pi \times 17^2 \times 17)/4(mm^3) = 3856.705mm^3$

Density=mass/ volume

1.12×10^{-3}= mass/(0.6×3856.705)

Mass of matrix= 2.519 g

Mass of epoxy= (90.9×2.519)/100

Mass of epoxy= 2.355g

Mass of hardener = (9.09×2.519)/100

Mass of hardener = 0.235 g

Mass of filler

Density of filler= mass/ volume

0.15×10^{-3}= mass/(0.40×3856.705)

Mass of filler= 0.231 g

In the similar manner calculation for all volume % of glass micro balloon was done and given in table:

Table 3.2 Mass of epoxy, hardener, and glass micro balloon for different volume fractions

Volume %	**Mass of epoxy (gm)**	**Massof hardener (gm)**	**Mass of glass micro balloon(gm)**
0	3.926	0.3926	0
5	3.7301	0.373	0.028
10	3.533	0.3533	0.0578
20	3.139	0.313	0.115
30	2.748	0.2748	0.173
40	2.355	0.235	0.231

3.4.3 Flexural test

Fig 3.6 Flexural test specimen placed on Instron machine

Modulus of rupture is the stress required to cause failure in bending. The test was performing on INSTRON machine with flexural test set up shown in figure 3.6.

Flexural strength= $(3pL/2bd^2)$

Where,

P = load,L=span length, b =breadth of sample, d =thickness of sample

Flexural test specimens were also calculated in similar manner as calculated in tensile test

3.4.4 Impact testing

Fig 3.7. Impact test setup, holding sample in groove.

Izod impact test setup supplied by **Jaya Electrical Electronics Scientific Equipments and Instruments** was used for conducting impact test. In this method determination of shock absorbing capacity is calculated for specimen under the shock loading condition. Before breaking of sample the energy absorbed was measured in a single hit. The standard specimen size is length (L) =63.5mm breadth (B) =12.3mm thickness (T) = 6.3mm.Impact energy can be defined as the energy requiredto fracture the sample by shock loading for small interval of time.

Calculation for impact test

Sample calculation for 0 volume %

The calculation for weight estimation for matrix and filler are as follow:

Calculation for matrix

Density of epoxy =1.15g/cc

Density of hardener =0.98g/cc

Density of glass micro balloon=0.15g/cc

Here epoxy to hardener ratio used was 10:1

Percentage of epoxy in matrix= (10/11) ×100=90.9%

Percentage of hardener in matrix= (1/11)×100=9.09%

By applying rule of mixture for obtaining density of matrix

Density of matrix= density of epoxy × volume fraction of epoxy+ density of hardener × volume fraction of hardener

Density of matrix= (90.9/100)×1.15+ (9.09/100)× 0.98

Density of matrix=1.12g/cc

Volume of mould =150×65×5mm^3 = 48750mm^3

Density= mass/volume

1.12×10^{-3}= mass/ (1×48750)

Mass of matrix= 54.6g

Mass of epoxy= (90.9×76.440)/100

Mass of epoxy= 49.63 g

Mass of hardener = (9.09×76.440)/100

Mass of hardener = 4.9 g

Sample calculation for 40 volume %

The calculation for weight estimation for matrix and filler are as follow:

Calculation for matrix

Density of epoxy =1.15g/cc

Density of hardener =0.98g/cc

Density of glass micro balloon=0.15g/cc

Here epoxy to hardener ratio used was 10:1

Percentage of epoxy in matrix= (10/11) ×100=90.9%

Percentage of hardener in matrix= (1/11)×100=9.09%

By applying rule of mixture for obtaining density of matrix

Density of matrix= density of epoxy × volume fraction of epoxy+ density of hardener × volume fraction of hardener

Density of matrix= (90.9/100)×1.15+ (9.09/100)× 0.98

Density of matrix=1.12g/cc

Volume of mould =150×65×5(mm^3) = 48750mm^3

Density= mass/ volume

1.12×10^{-3}= mass/(0.6×48750)

Mass of matrix= 32.760g

Mass of epoxy= (90.9×45.864)/100

Mass of epoxy= 29.77 g

Mass of hardener = (9.09×45.864)/100

Mass of hardener = 2.97 g

Mass of filler

Density of filler= mass/ volume

0.15×10^{-3}= mass/(0.40×48750)

Mass of filler= 2.925 g

In the similar manner calculation for all volume % of glass micro balloon was done and given in table:

Table 3.4Contains mass of epoxy, hardener, and glass microballoon

Volume %	Mass of epoxy (gm)	Massof hardener (gm)	Mass of glass micro balloon (gm)
0	49.631	4.96	0
5	47.149	4.71	0.36
10	44.668	4.466	0.7312
20	39.705	3.970	1.462
30	34.74	3.47	2.1937
40	29.77	2.97	2.925

3.4.5 Hardness test

Vickers hardness test consist of indentation of test material with diamond indenter in the form of pyramid with square base and angle was 136^0. Load is normally applied for 10-15sec.The two diagonal surface of indentation after removal of load was measured by in situ microscope and their average calculated. The test was performed on LECO Vickers hardness tester LV700.The unit of Vickers hardness we take in HV as per the following equation-

$HV=1.854(F/d^2)$

Where,

F=load applied, d =mean of diagonals of indent

By multiplying hardness value (HV) to 10 it converted to MPa

3.5 Tribological characterization

3.5.1 Erosion test

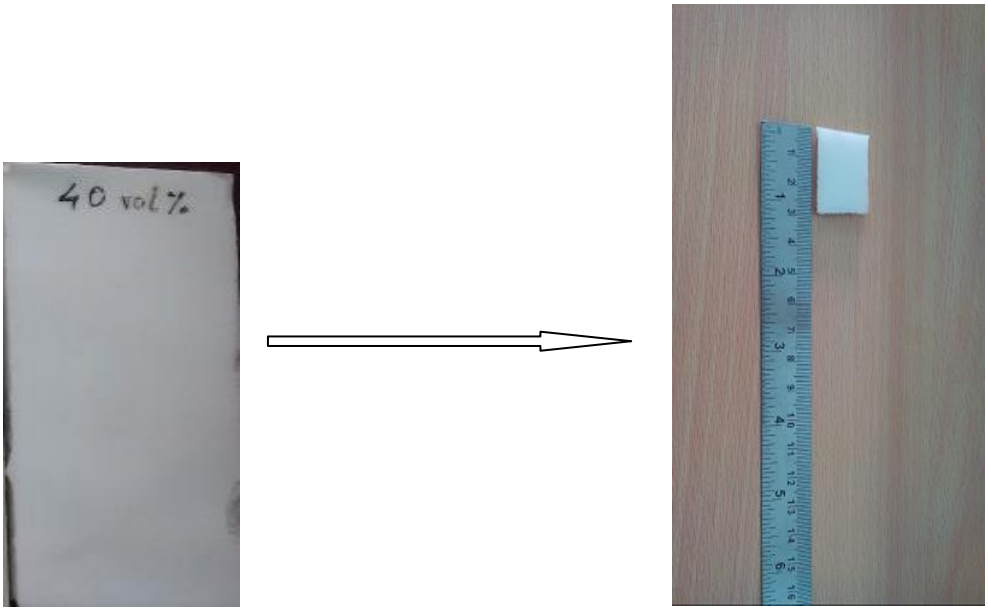


Fig 3.8 sample for erosion test

Erosion test was performed as per ASTM G-76 standard and perform on machine MAGNUM PVT LTD. samples were cut in the dimension 25×25×6 mm^3 from the as cast composite slab. The erosion set up (as shown in fig 3.9)consists of air compressor, a particle feeder, nozzle, and pulley. Flow rate was maintained at $2gmin^{-1}$for the present study and erosion time 15 minutes (five equal intervals). The stand-up distance from nozzle to sample is 10mm. The sand particles use as an erodent material. The erosion rate for each volume % of syntactic foam was measured as a function of three different velocities (48, 70, 82 m/s) and three different angles (30^0, 60^0, 90^0) respectively.

Figure 3.9-Shows 30^0 sample in erosion machine

Calculation for erosion rate(E_r)

Erosion rate can be defined as the ratio of volume loss of specimen material to the total mass of the erodent impact on it [18].

E_r= (W_i- W_f) / (total mass of erodent impact) ……(3.5)

W_i=initial mass of the sample, W_f = final mass of the sample

Total mass of erodent impact=erodent flow rate(gm/min)× (3 minutes for each impact) ×5 times impact

Total mass of erodent impact =2 gm min^{-1}× (3 min ×5)

Total mass of erodent impact = 30 gm (constant for every sample)

Sample calculation for 0 volume %, impact velocity 48m/s, at 90^0

W_i =3.555 gm, W_f =3.544gm

Flow rate for erodent = 30 gm

Put the given values in equation (3.5)

E_r= (3.555-3.544) / 30

E_r=0.00036 gm/gm

Chapter 4

Result and Discussion

In this chapter, results of various physical characterizations of glass microballoons are described first. It is followed by the discussion on mechanical and erosion properties of the syntactic foam.

4.1Physical testing

4.1.1 Particle size analysis of glass microballoon

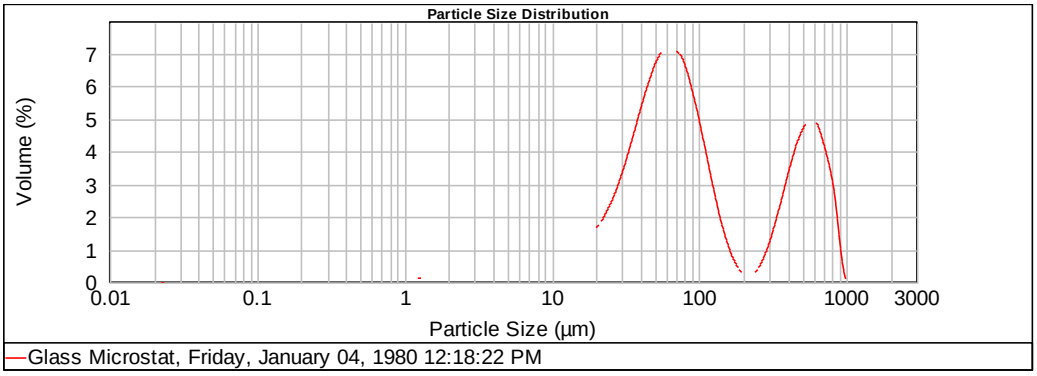


Fig 4.1 Particle size distribution of glass microballoon

From the particle size analysis of glass microballoon, it is seen that the minimum particle size of glass microballoon was 19.01µm, maximum particle size of glass micro balloon is 601.15 µm and average particle size was 71.452 µm. It is also seen from the graph that maximum volume % of glass micro balloon found between 10 µm to 100 µm.

4.1.2 SEM and EDS of glass micro balloon

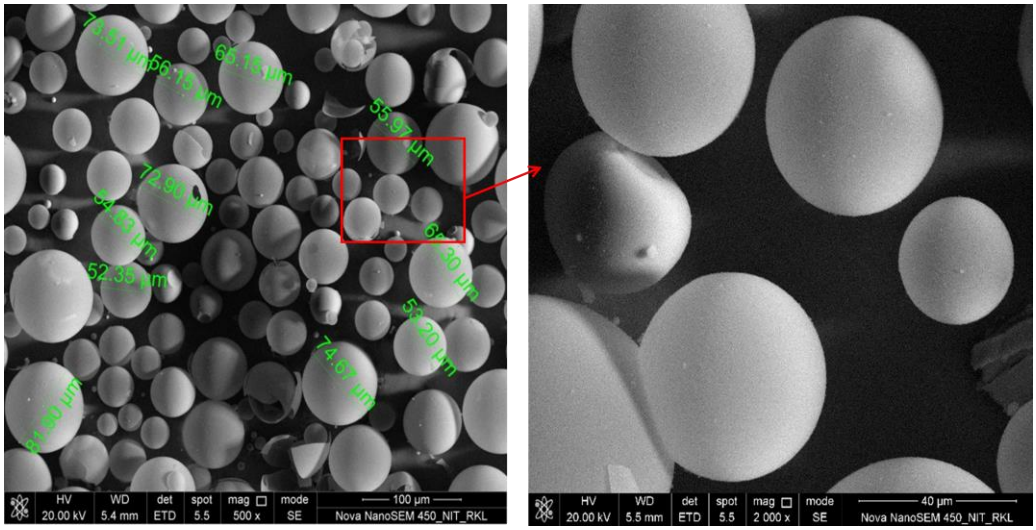


Fig 4.2-SEM image of glass micro balloon

Glass microballoon is having spherical morphology, as evident from the above figure.

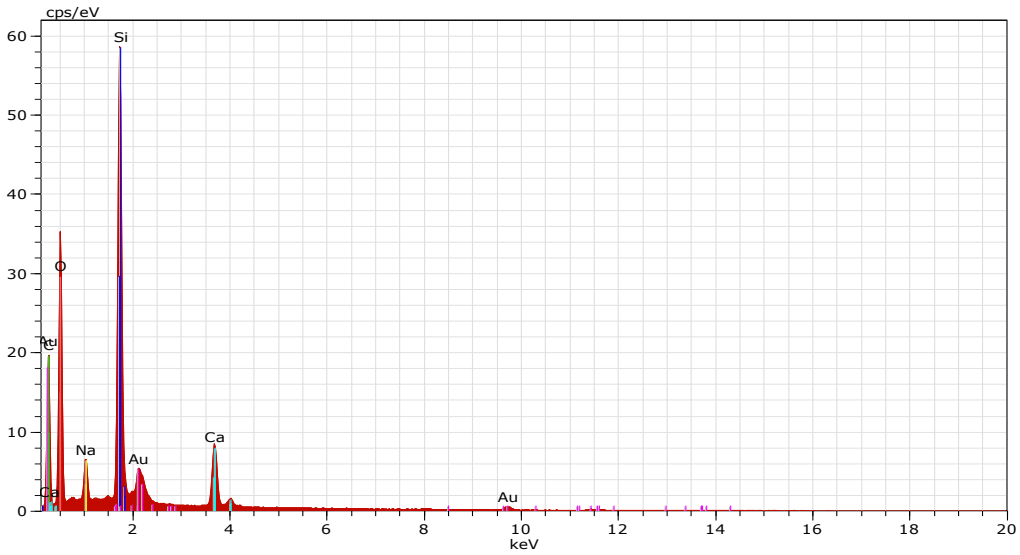


Fig 4.3- EDAX of glass microballoon

Chemical composition of glass microballoon is soda lime borosilicate and this composition was supported energy discharge spectroscopy of glass microballoons particles.

4.1.3 XRD of glass microballoon

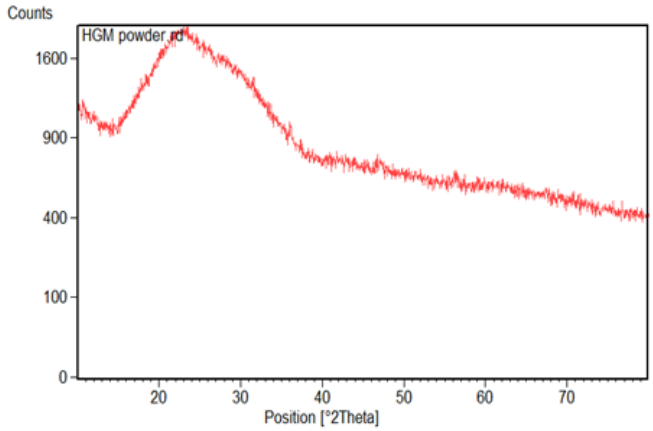


Fig 4.4 XRD of glass microballoon

In present study, hollow glass microballoon was used as reinforcement. Its amorphous nature is confirmed by XRD (shown in the above figure 4.4).

4.1.4 Density measurement of syntactic foam

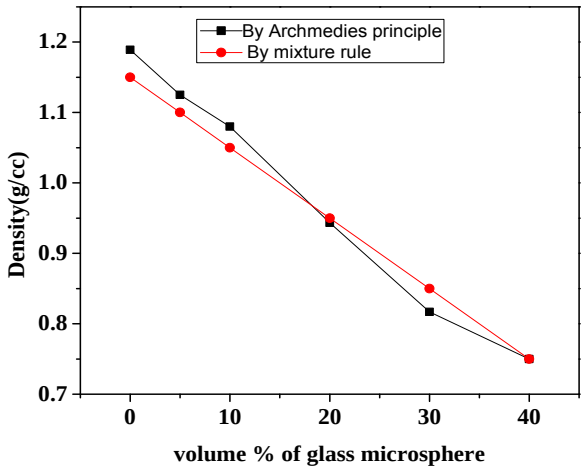


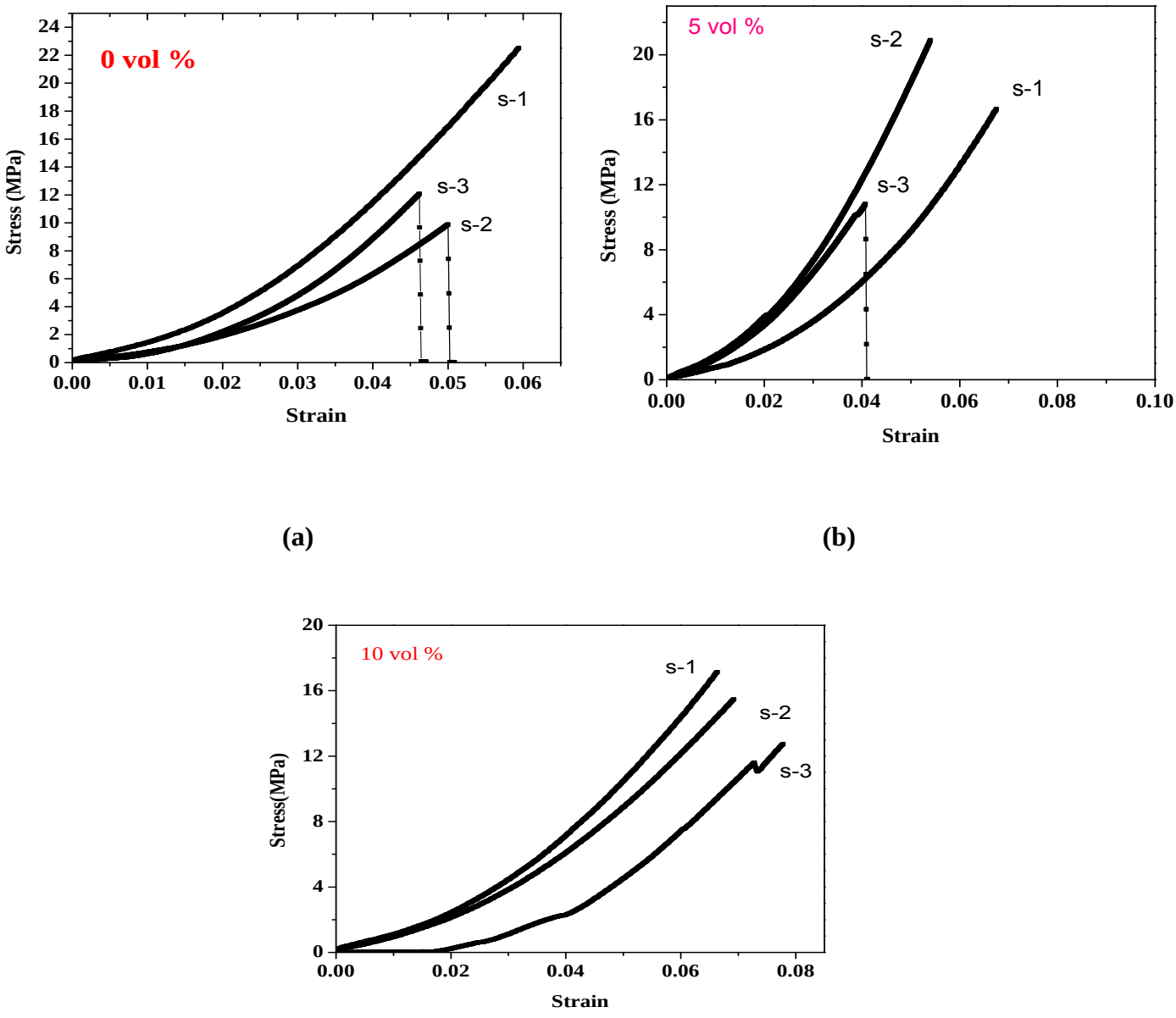
Fig 4.5- variation of density with varying volume % of glass micro sphere

Density was calculated by two methods first by rule of mixture and second by Archimedes principle as shown in above graph. As the volume % of glass microballoon increases density of syntactic foam decreases because density of glass micro balloon was 0.15g/cc, by adding glass micro balloon effect of balloon will be more as compared to neat epoxy.

4.2 Mechanical testing

4.2.1 Tensile testing

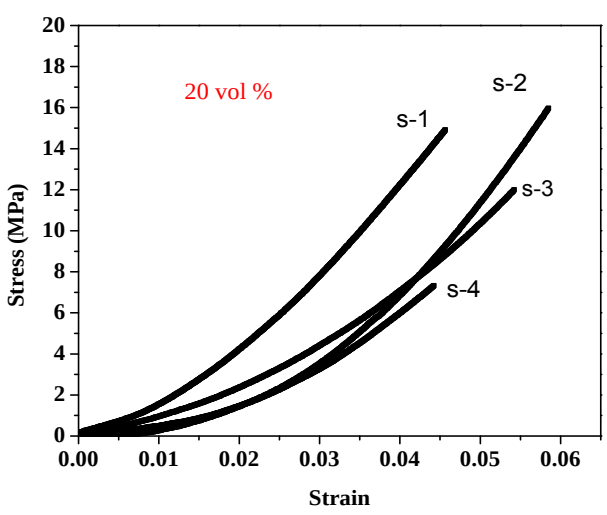
Tensile stress- strain curve of the syntactic foam with different volume percentage of glass microballoon are shown below



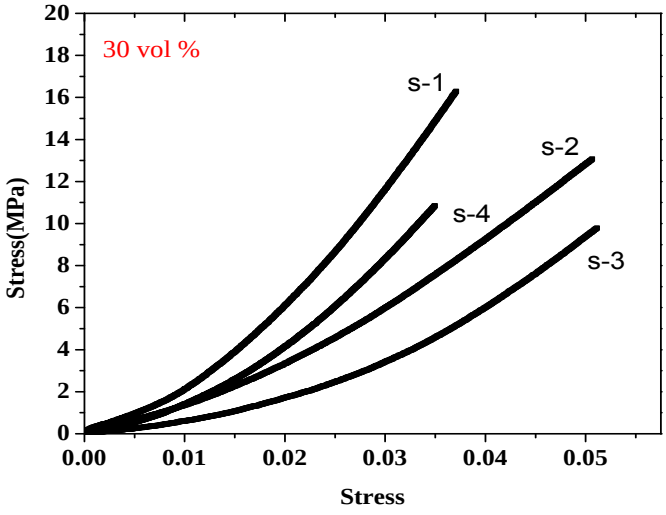
(a) **(b)**

(c)

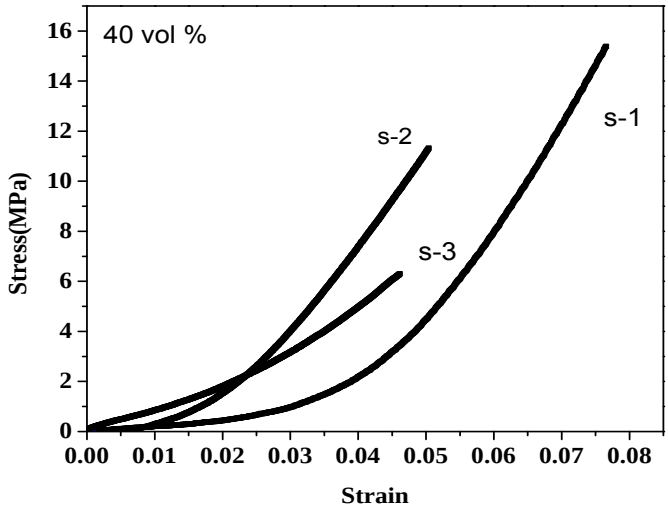
Fig 4.6 (a) stress-strain graph for 0 volume %, (b) 5 volume %, (c) 10 volume %



(a)



(b)



(c)

Fig 4.7 (a) stress-strain curve for 20 volume %, (b) 30 volume %, (c) 40 volume %

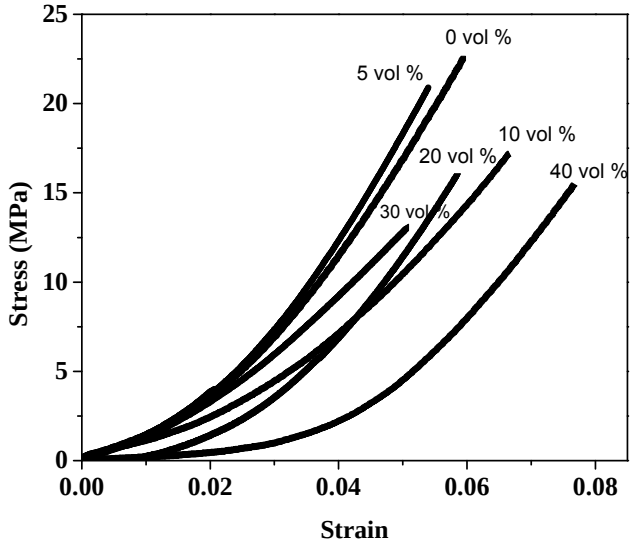


Fig 4.8- variation of stress with strain for all volume percentages

With increases in volume percentages of glass micro balloon, interfacial layer between epoxy and glass microballoon decreases and resulting decreases in stress value from 22MPa for neat epoxy to 13MPa shown in above figure4.8

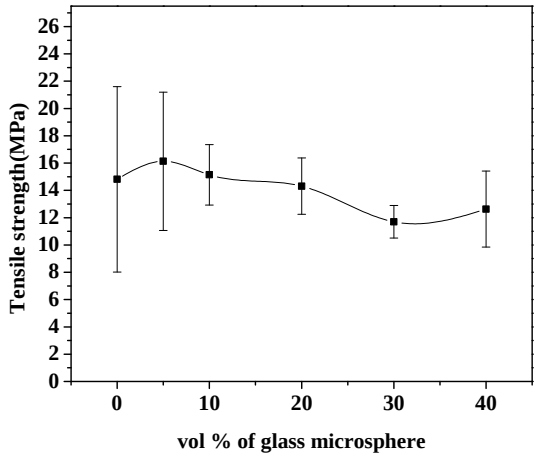


Fig 4.9- variation of tensile strength with volume % of glass micro sphere

Tensile strength of epoxy/glass micro balloon syntactic foam increasing from 0 volume % to 5 volume % and then decreasing, this is due to the fact that with inclusion of glass micro balloon reduces the volume fraction of epoxy results poor bonding between epoxy resin and glass microballoon.

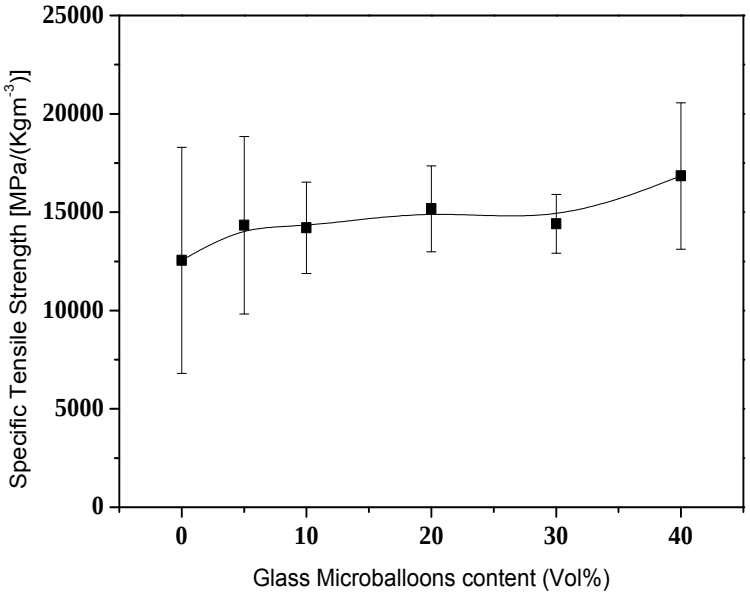


Fig 4.10- Specific tensile strength with varying glass microballoon content

After replacing epoxyresin with 40 volume% glass microballoon,density of syntactic foam is reduced by 38%, but specific tensile strength of syntactic foam composite is increased by 34%. This is desirable for using this type of syntactic foam for light weight structural applications.

Note that Mean and standard deviation is based on 3-4 samples in each set (i.e. volume %)

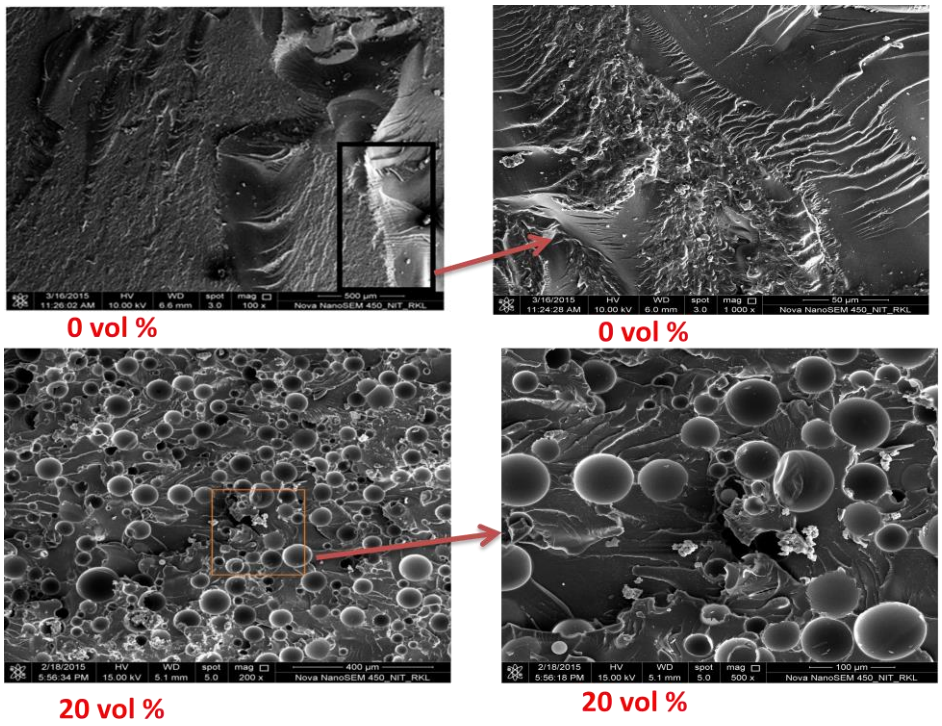
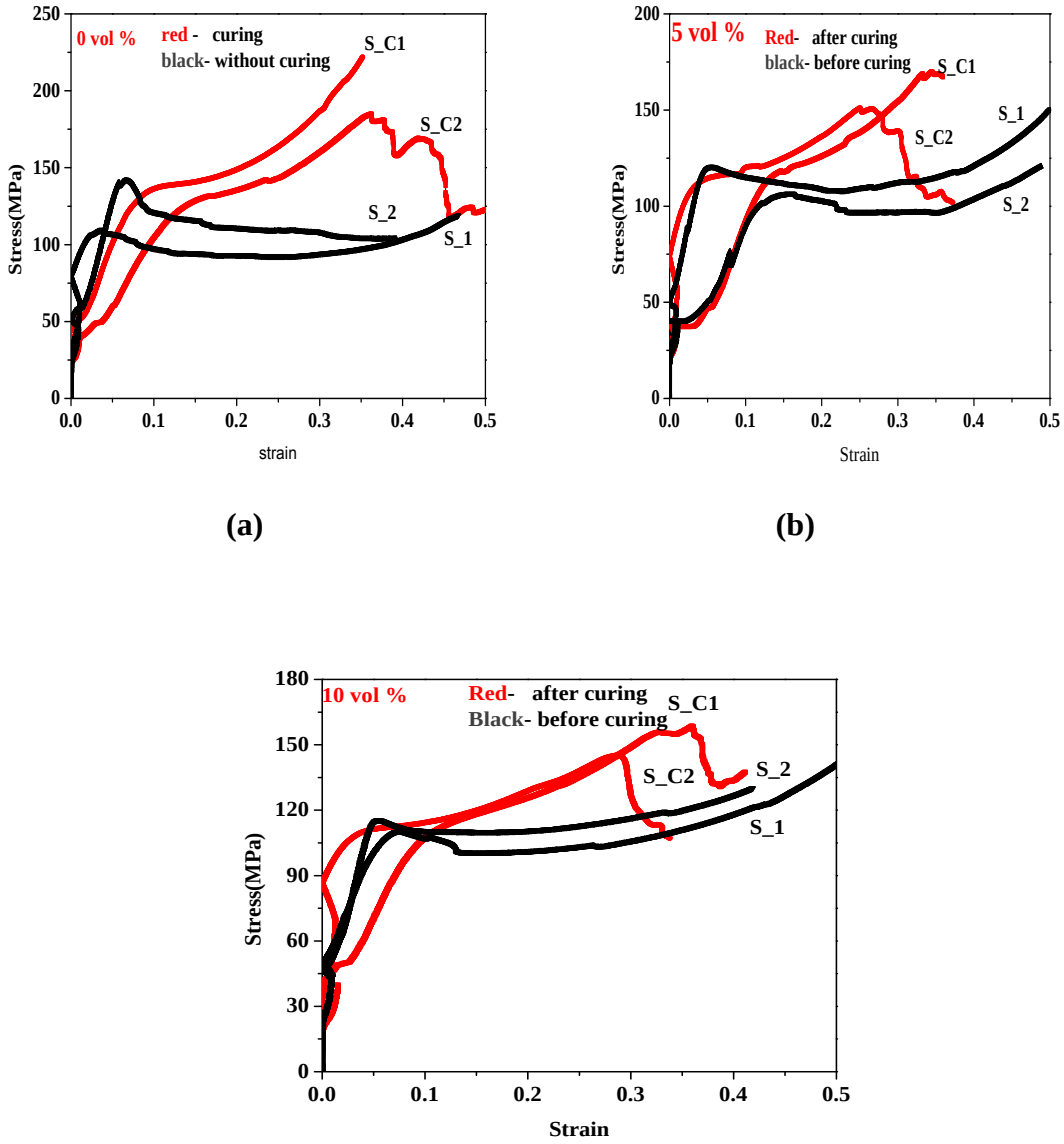


Fig 4.11- SEM images of syntactic foam for 0 volume % and 20 volume %

During tensile loading, particles-matrix debonding occurs. From the fracture surfaces, it is evident that deformation under tensile loading mostly occurs in the matrix while glass microballoon mostly remains intact after deformation.

4.2.2 Compression test

Compression stress- strain curve of the syntactic foam with different volume percentage of glass microballoon are shown below.



(a) **(b)**

(c)

Fig .4.12 Stress-strain curve for (a) 0 volume % (b) 5 volume % (c) 10 volume%

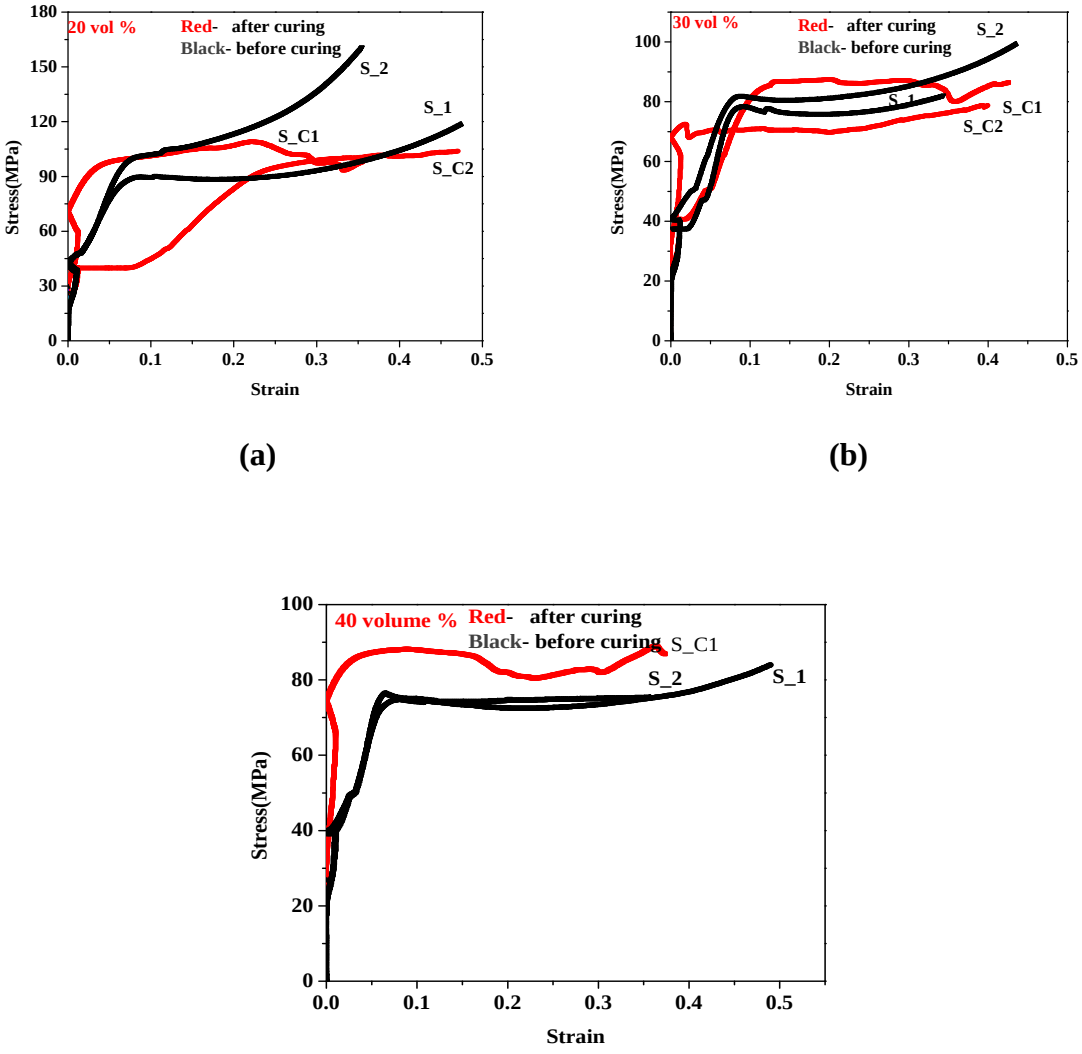


Fig .4.13 stress-strain curve for (a) 20 volume % (b) 30 volume % (c) 40 volume%

The stress- strain curve can be categorized in three region (i) linear elastic region (ii) plateau region and (iii) densification region as shown in figure 4.12. In elastic region material is subjected to uniform deformation and a linear elastic region form. Stress value reaches to maximum value by increasing load and then attains a constant value which indicates plateau region. The maximum value of the stress denotes the initiation of crack in the pure resin or foams. After the crack formation plateau region start, where the continuous deformation of material takes place. The plateau region corresponds to breaking of microballoon that opens the enclosed space (within the microballoons) and providing more space for compressing. When the significant amount of glass microballoons are crushed, stress level starts increasing

which is the indication of process of densification of the material and more amount of plastic deformation in the matrix.

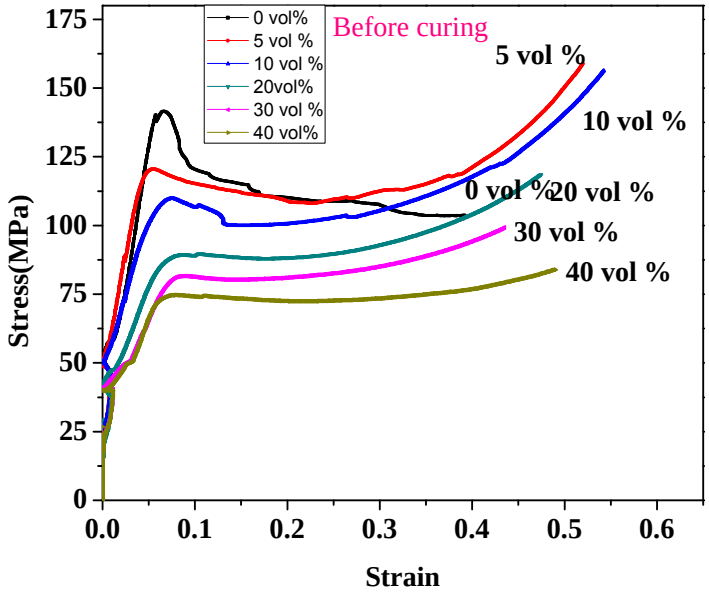


Fig 4.14-stress strain relation for compression test curing sample at room temperature

Hollow glass micro balloon used in the study are brittle in nature having low crushing strength of 2MPa in contrast to the epoxy 140MPa. Increasing the amount of glass micro balloon obvious the epoxy resin decreases and leaving a thin interfacial layer between epoxy resin and glass microballoons. As a consequence, decrease in the load bearing capacity of matrix phase, and the compression strength of syntactic foam shows decreasing trend.

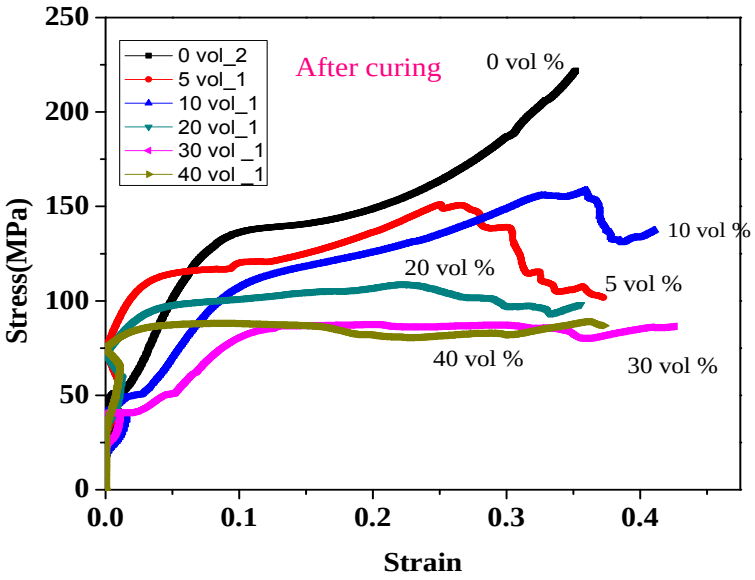
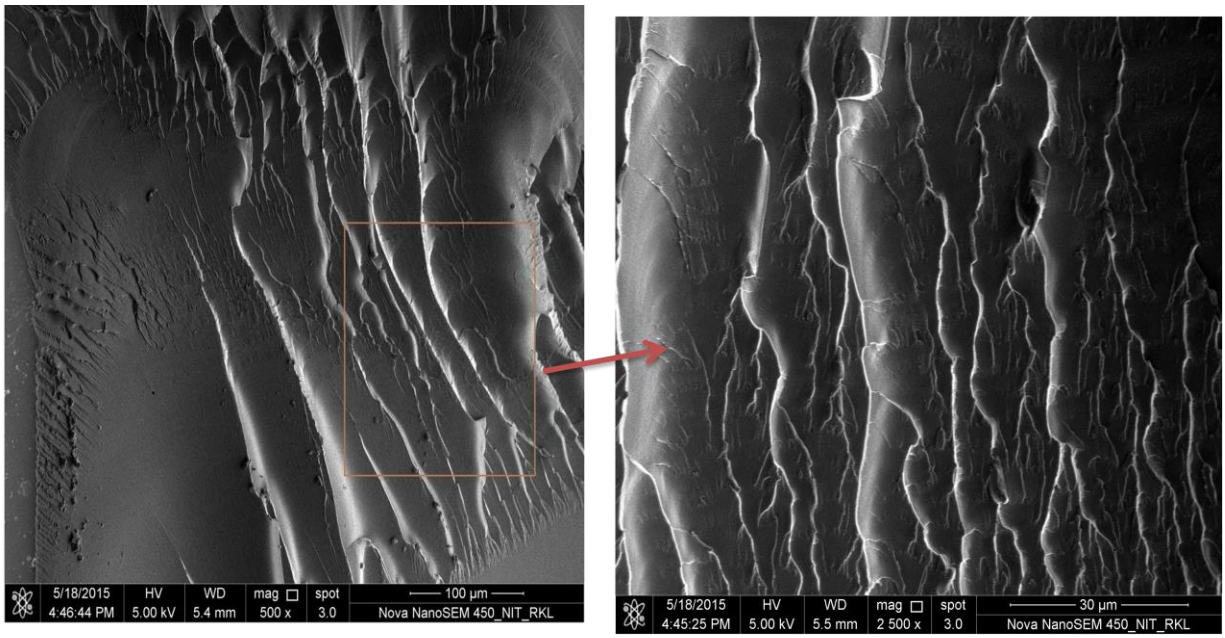


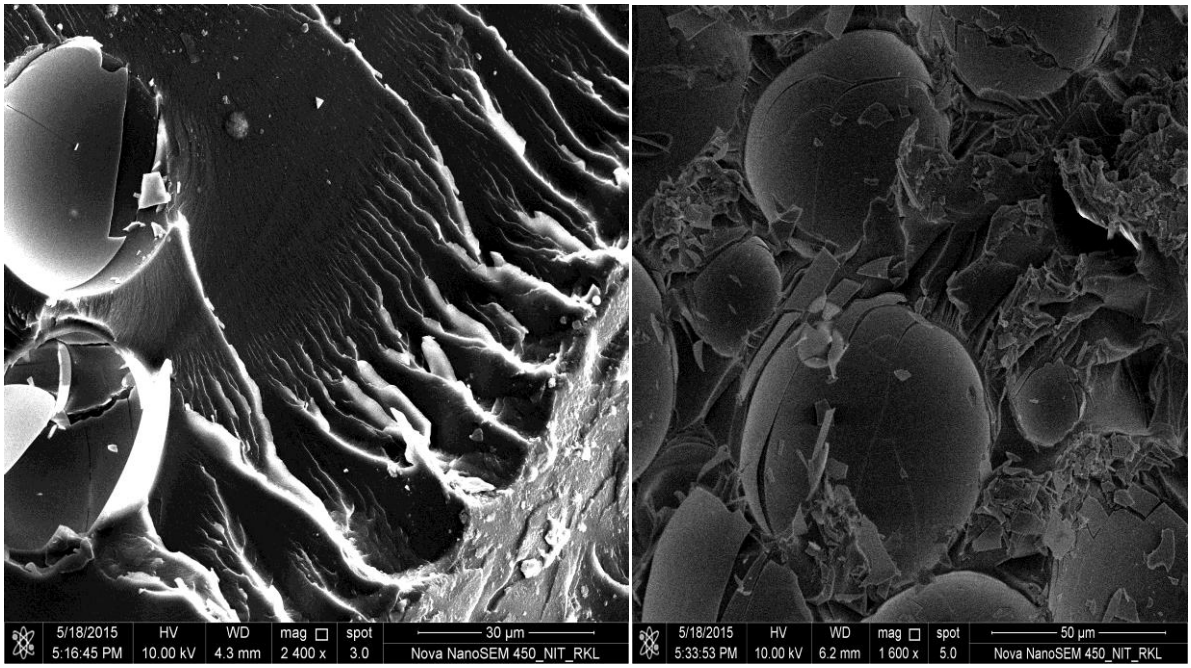
Fig 4.15-stress strain relation for compression test after curing

After testing on sample cured in room temperature, test performed on the sample cured at 120^0C for 4 hours. After testing it was seen that the compression strength of sample increasing as compared to curing at room temperature.



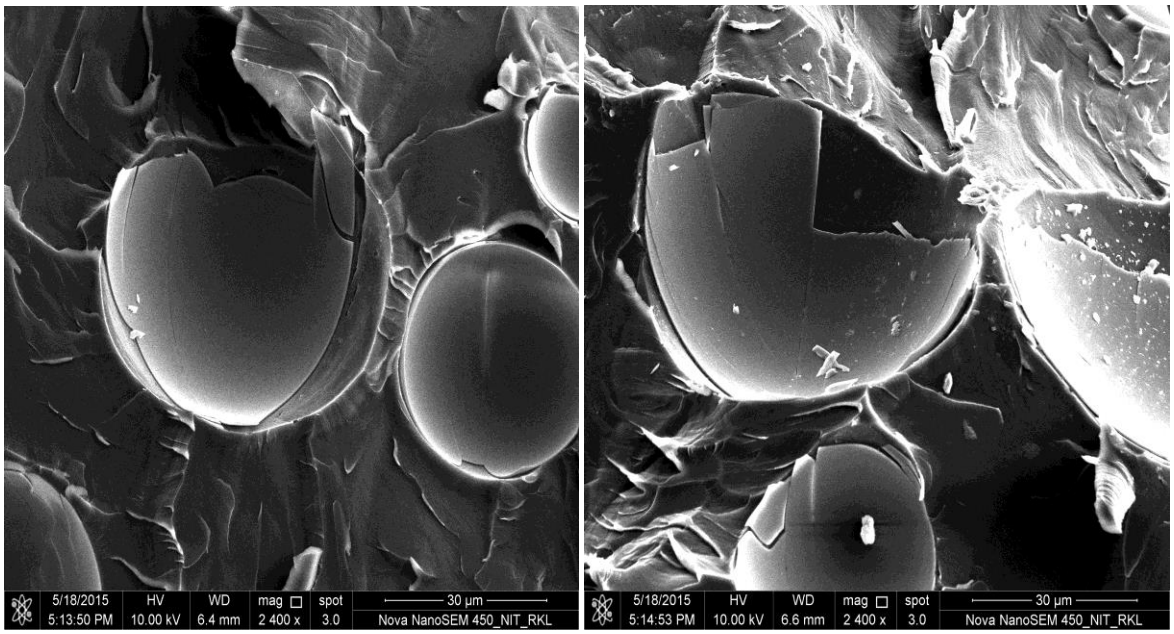
(a) SEM of 0 volume % at 500 magnification (b) SEM of same sample at 2500 magnification

Fig 4.16 - SEM images for 0 volume % at higher as well as lower magnification



(a) **(b)**

Fig 4.17-(a) SEM image showing crack arresting of 10 volume % (b) SEM image showing crushed microballoon for 40 volume%



(a) **(b)**

Fig 4.18 .Crushed glass microballoon for 10 volume percentage

4.2.3 Flexural test

Flexural strength curve of the syntactic foam with different volume percentage of glass microballoon are shown below.

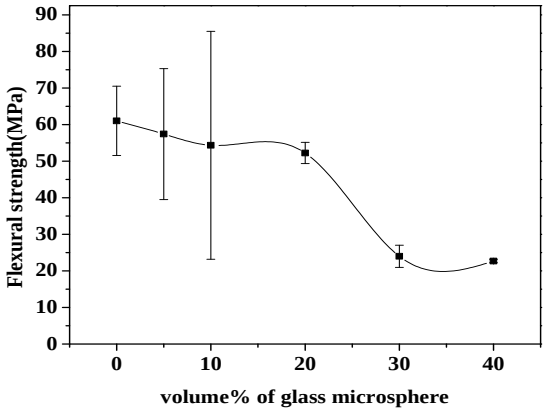
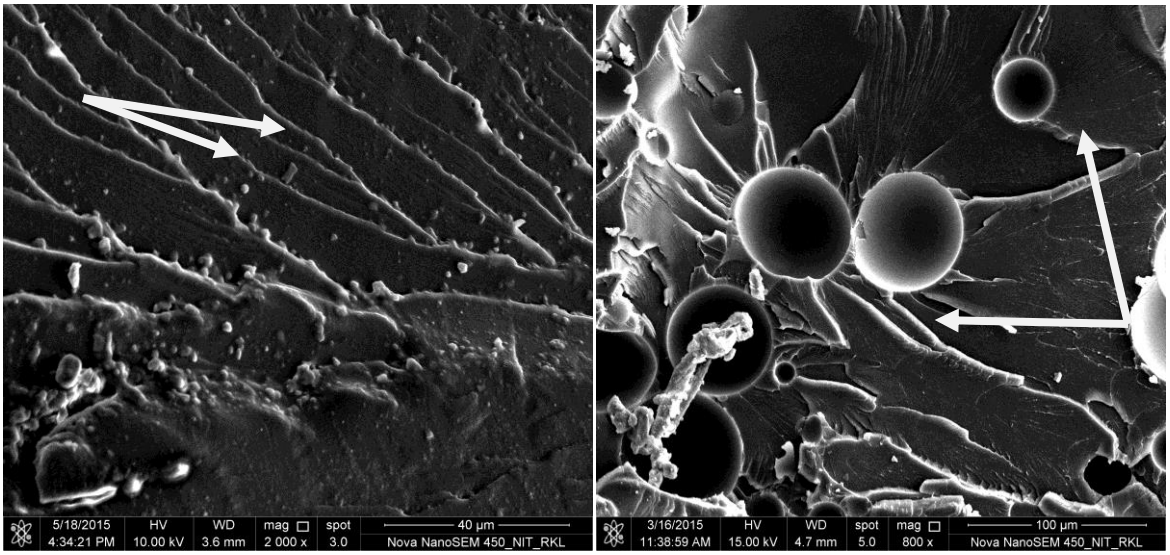


Fig 4.19 Variation of flexural strength with volume % of glass micro balloon

Flexural strength of the epoxy glass micro balloon syntactic foam decreases with increasing in volume % of micro balloon as shown in figure. The reason for trend was due to vacuole formation, as the volume % of glass micro balloon increases vacuole growth will also occur. The vacuole in flexural test was developed on tension side of sample.



(a) (b)

Fig 4.20 (a) SEM image for 0 volume % (b) SEM image for 40 volume %

Above figure shows SEM image for flexural test specimen (a) shows the wrinkles developed in the test specimen in 0 volume % sample denoted by arrow, (b) this figure shows two phenomenon crack arrested by glass micro balloon and crack divergent by glass micro balloon.

4.2.4 Impact testing

Impact strength curve of the syntactic foam with different volume percentage of glass microballoon are shown below.

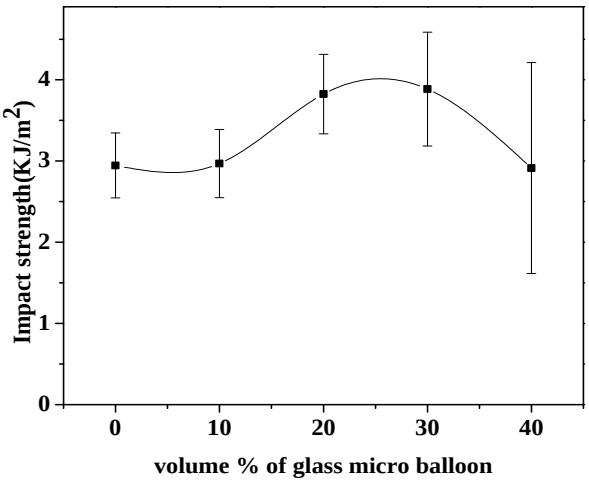
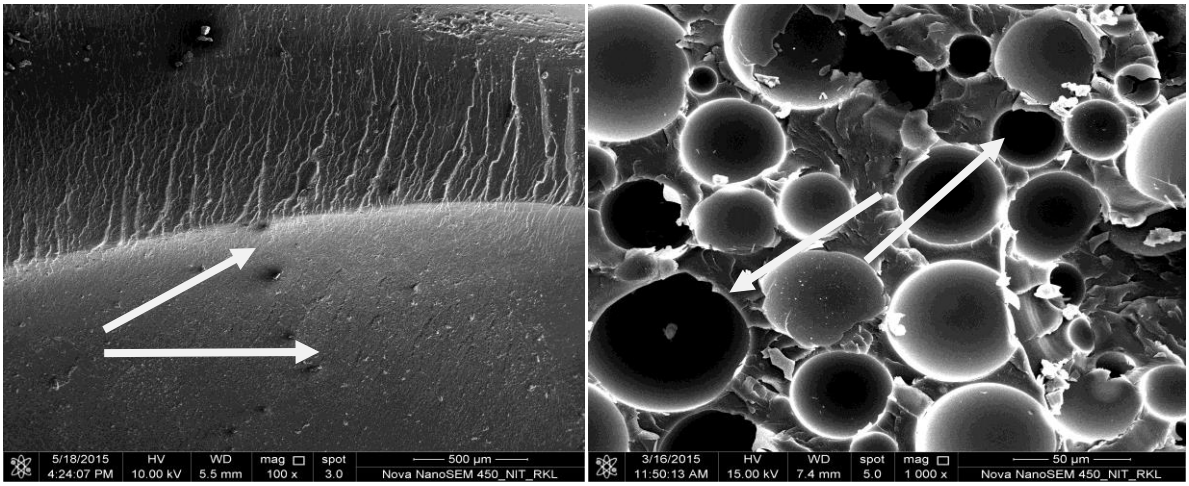


Fig 4.21- Variation of impact strength with volume % of glass micro balloon

Above figure shows variation of impact strength by varying volume % of glass micro balloon. Impact strength of sample increasing with increase in volume percentage of glass micro balloon up to 20 volume % and remain almost same for 30 volume % and then start decreasing. The reason for this trend is that with increase in volume % of glass micro balloon the bonding between epoxy and glass micro balloon bears good strength, but after a certain value the interface between epoxy and glass micro balloon become thin result in decrease impact strength.



(a) (b)

Fig 4.22 (a) SEM image of 0 volume % (b) SEM image of 40 volume %

SEM image for impact test are shown in figure-(a) the image for 0 volume % shows two things, area where strain developed in the sample and the area without strain. In impact test sample break in a fraction of second means it gives no time to the sample to develop strain in it, this phenomenon is supported by figure (a) because we see less strain in the sample. Figure-(b) shows the SEM image of 40 volume % sample, the pull out of glass micro balloon from the matrix can be seen from the image denoted by arrow.

4.2.5 Hardness test

Hardness curve of the syntactic foam with different volume percentage of glass microballoon are shown below.

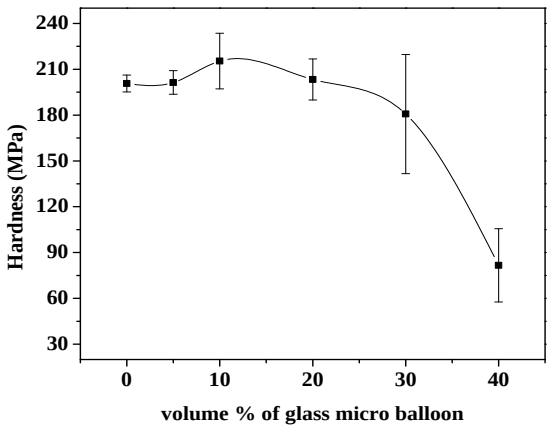
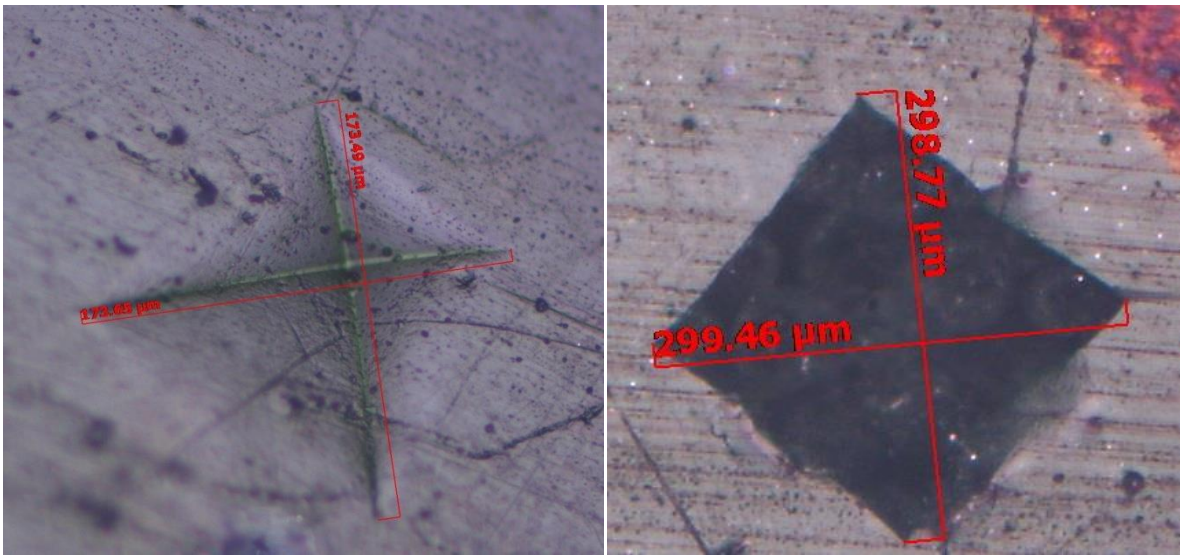


Fig 4.23 Variation of hardness with volume % of glass micro balloon

From the figure it is seen that hardness value increases with increase in volume fraction of glass micro balloon up to an optimum limit and then start decreasing. This is due to the cluster size of glass micro balloon reaches a crucial limit and then reinforcing function of glass micro balloon start decreasing; resulting decrease in hardness value of syntactic foam.



(a) (b)

Fig 4.24 (a) microscopic image of 0 volume % (b) microscopic image of 5 volume %

Above figure shows the microscopic image for hardness test specimen (a) shows the image for 0 volume % of a sample with indenting diagonals. (b) Microscopic image of 5 volume % sample with indenting diagonals

4.2.6 Erosion test

Erosion rate curve of the syntactic foam for different angles are shown below.

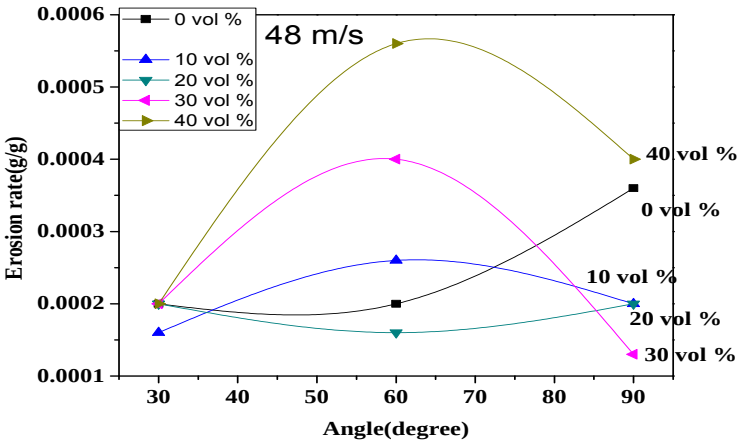


Fig 4.25 Variation of erosion rate with different angles for impact velocity 48 m/s

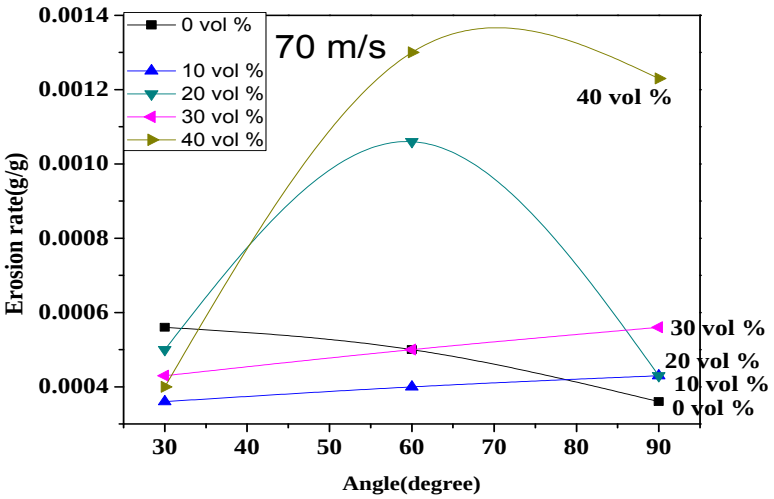


Fig 4.26 Erosion rate and angle graph for 70 m/s impact velocity.

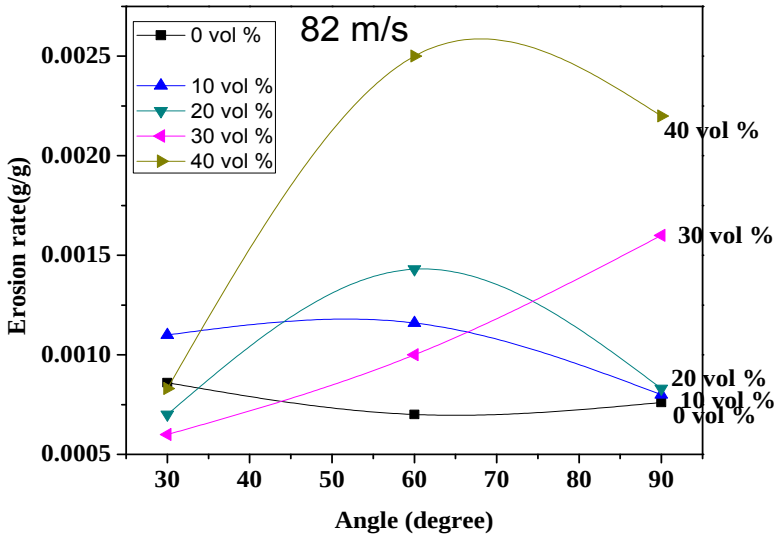


Fig 4.27 Variation of erosion rate with angle at 82 m/s impact velocity

By considering 90^0 angle and impact velocity 82 m/s, it is found that erosion rate increasing from 0 volume % to 40 volume % as shown in figure, because for lower volume percentage epoxy content is more and by increasing volume percentage hollow glass microballoon content increasing and it will come in exposure to sand particle more as compared to epoxy. And also crushing strength of glass microballoon is less as compared to epoxy so erosion rate increases.

Table 4.1- variation of erosion rate for different volume % and at different impact velocities and angles

Erosion rate (low to high) of different volume %	Impact velocity								
	48 m/s			70 m/s			82m/s		
	30^0	**60^0**	**90^0**	**30^0**	**60^0**	**90^0**	**30^0**	**60^0**	**90^0**
	10	20	30	10	10	0	30	0	0
	0,20,30	0	10,20	40	0,30	10,20	20	30	10
	40	10	0	30	20	30	0,40	10	20
		30	40	20	40	40	10	20	30
		40		0				40	40

Considering, erosion at 82 m/s impact velocity and 90^0angles because glass microballoon is light in weight and it is possible that in syntactic foam glass microballoon will float over epoxy and true wear of foam will not measure as shown in schematic diagram in figure, so in 30 for all impact velocity and 48m/s impact velocity is neglected.

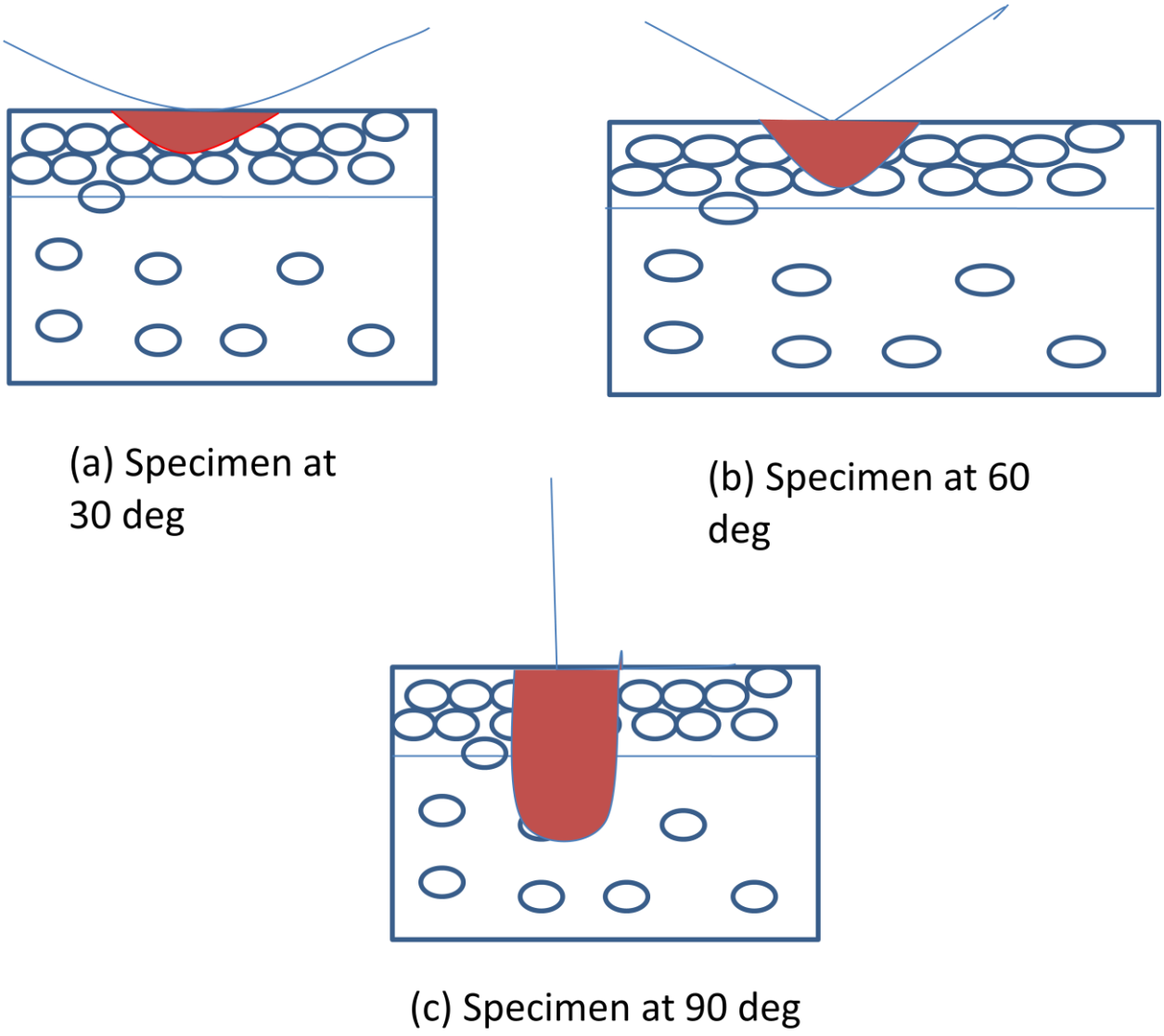


Fig 4.28 Effect of erodent at different angles

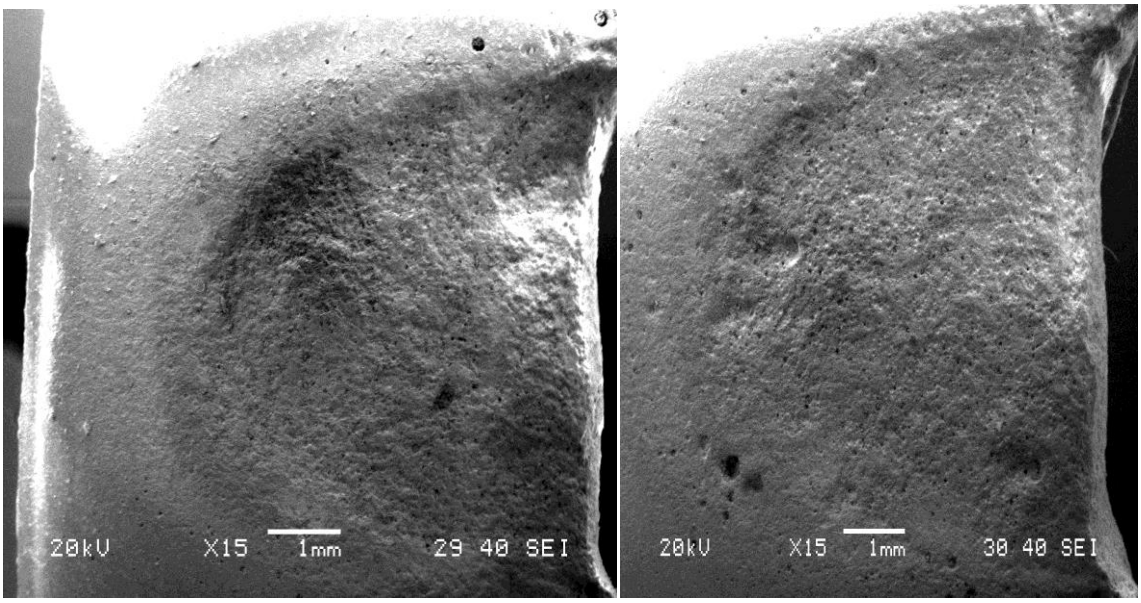
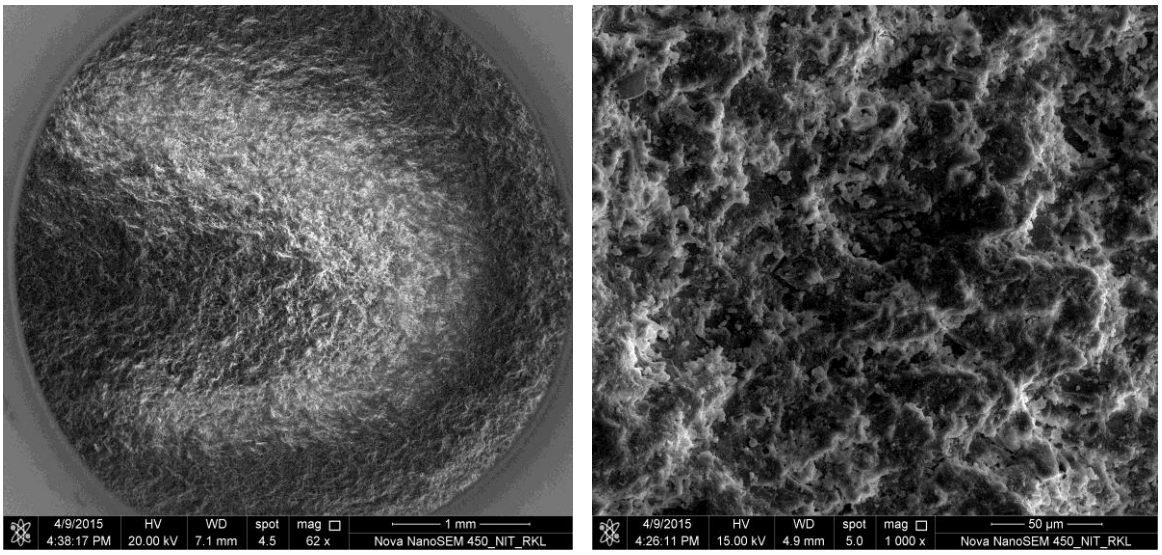
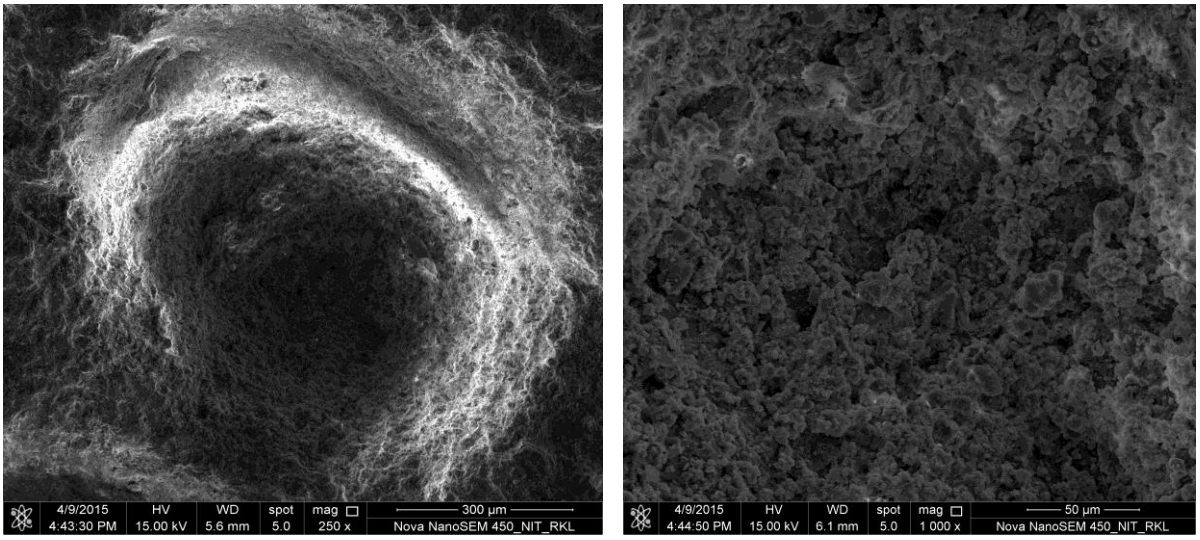


Fig 4.29 (a)SEM image of 40 volume % sample for 82m/s impact velocity at 30^0 (b) SEM image of 40 volume % for 70m/s impact velocity at 30^0.



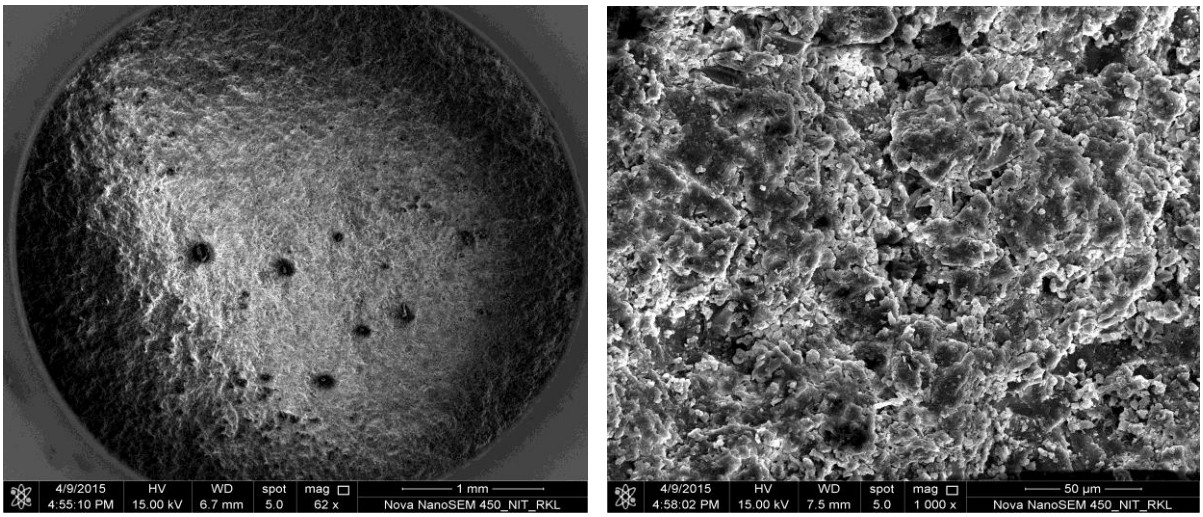
(a) (b)

Fig 4.30 SEM image 0 volume % and 30^0 (a) lower magnification (b) higher magnification



(a) **(b)**

Fig 4.31 SEM image 0 volume % and 60^0 (a) lower magnification (b) higher magnification



(a) **(b)**

Fig-4.32 SEM image 0 volume % and 90^0 (a) lower magnification (b) higher magnification

Sample eroded maximum for 90^0 and 82 m/s as because epoxy resin glass micro balloon syntactic foam was brittle in nature and brittle sample eroded more for 90^0 and ductile sample eroded more for 30^0 shown in figure and minimum for 30^0 and 48 m/s.

Chapter 5

Conclusion

The present experimental study on synthesis and mechanical properties of epoxy resin matrix based syntactic foam with varying volume percentages of hollow glass microballoons reveals that,

- The thermosetting resin (epoxy) matrix based syntactic foam with different volume percentages (0-40 vol%) of glass microballoons was successfully prepared by stir casting method. Beyond 40 vol.% of hollow microballoons, it was not possible to process the foam mixture due to very high viscosities.

- Although the tensile strength of the syntactic foam was decreased from 14 MPa (pure epoxy resin) to 12 MPa (40 vol.% of reinforcement), the *specific* tensile properties of the syntactic foams are higher than those of the neat resin owing to the lower densities of the foams. Shear yielding of the resin matrix followed by debonding at the matrix-microballoons interfaces may be the reason for fracture during tensile deformation.

- The compressive strengths of the syntactic foams decreased (from 140 MPa to 75 MPa) with the increase in volume fraction of the glass microballoons (up to 40 vol.%). The deformation behavior of syntactic foam is similar to that of cellular solids; crushing of the microballoons and simultaneous densification of the matrix may believe to be the deformation mechanism during compression; vindicated by the microstructural studies of the fractured surfaces. Thermal curing at 120^{o}C for 4 hours was found to be effective in increasing the compressive strength of the syntactic foam.

- With the increase in microballoon content, energy absorption capacity of the syntactic foam increases from 3 kJm^{-2} (for pure resin) to 3.8 kJm^{-2} up to a critical volume fraction (30 vol.%).

- Erosion rate was found to be maximum for 40 vol.% foam corresponding to the erodent velocity of 82 m sec^{-1} and impingment angle of 90^{o}. The segregation of lower density glass microballoon to the upper surfaces of the as-cast samples during processing may be responsible for highest erosion rate.

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